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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL MEMORANDUM 1384

METALLOGRAPHY OF ALUMINUM AND ITS ALLOYS USE OF ELECTROLYTIC POLISHING

By P. A. Jacquet

Translation of "Métallographie de l'aluminium et de ses alliages. Emploi du polissage électrolytique." Office National d'Études et de Recherches Aéronautiques, Publication No. 51, 1952.



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SUMMARY

This report has the purpose of:

(1) Furnishing bibliographic references of work done during the last 3 years regarding the physical chemistry of aluminum and its alloys, using electrolytically polished surfaces.

(2) Presenting some data on modern micrographic techniques (preparation of specimens, anodic oxidation, etch figures) which also are based on electrolytic polishing.

(3) Describing the use of an automatic apparatus for electrolytic polishing, suitable for obtaining micrographic specimens of aluminum and all its alloys, of homogeneous or polyphase structure.

The whole constitutes a report of purely practical character which should permit the metallographer of metals and light alloys to choose the technique most appropriate to his purpose. The information contained in our report on electrolytic polishing (published 1948 at Editions Métaux "Le polissage électrolytique des surfaces métalliques et ses applications, Tome 1. Aluminium, Magnésium, Alliages légers") is assumed to be known.

INTRODUCTION

The technical developments of light and ultralight alloys require more and more the use of precise and rapid control methods. Laboratory studies for the perfection of new materials possessing interesting qualities also are based on examination procedures which include, in particular, ordinary and electron micrography. In this last case, the quality of the photomicrographs obtained is of primary importance; however, the speed

*"Métallographie de l'aluminium et de ses alliages. Emploi du polissage électrolytique." Office National d'Études et de Recherches Aéronautiques, Publication No. 51, 1952.

in preparation of the specimens is not to be overlooked because it permits both a reduction in subordinate personnel and an increase in the researchers' output.

The almost exclusive use of electrolytic polishing in micrography which has replaced the formerly used mechanical procedure has, in a general manner, achieved a considerable improvement of metallography. Its value is even greater for aluminum and its alloys, in view of the difficulties of a correct mechanical polishing which are well known to the specialists.

The complete reduction of superficial irregularities and the improvement in quality of the surfaces prepared with the methods of electrolytic polishing have, moreover, led to entirely new possibilities which widen the employment range of the microscope. Thus, micrography is no longer limited to routine observation of grain shape and size or to detection of the different phases that might be present.

The films of oxides, the etch figures, the microhardness, the phase contrast, the interferences, the actual developments of which are due to a large extent to electrolytic polishing, give us the means to study in detail structure and texture of light materials in various states, and to investigate the mechanisms of the fundamental processes such as cold plastic deformation, fatigue, creep, crystallization, aging, etc.

In order to profit from these new methods, the metallographer must familiarize himself with the technique of electrolytic polishing which depends more on chemistry than on his specialty. Thus, it may happen that the preparation of specimens might be entrusted to employees who are not always well aware of the result to be obtained and might lack, as well, the means and the time for adapting a polishing technique to a determined aim.

On the other hand, the improvements and methods of realization published in the world literature do not always become known to the men who could derive an immediate profit from or be stimulated by them.

The object of the present report is to remedy these difficulties as far as possible, that is, to offer to metallurgic laboratories specialized in metals and light alloys the most recent data on the composition, the procedures, and the practical results of modern electrolytic polishing baths.

We have already treated the methods of electrolytic polishing applicable to common metallic materials in a previous report. Another report contained a general review of all scientific applications. Aside from the fact that both are now no longer up to date, the magnitude of the subject forbade an entering into the details of which the experimentator, however,

has the greatest need. One will find here the elements for closing these gaps. Of course we assume as known (and consequently shall not take into account) the historically oldest polishing procedures as described in our aforementioned report. Nevertheless we shall make an exception regarding certain techniques to the extent to which they have served as a basis for notably improved results.

Our report will be divided into three parts. In the first, we give the complete list of bibliographic references concerning metallographic studies which have, at whatever stage, required employment of electrolytic polishing. All these references apply to reports published later than 1947 and thus do not appear in the general report mentioned above. They have been arranged according to the composition of the electrolytes used in the polishing when the authors specify them.

The second part describes several general micrographic techniques including, besides polishing proper, the methods of anodic oxidation, of etch figures, and of thinning specimens for examinations by X-rays and electron microscope.

The third part presents an unpublished experimental study on electrolytic polishing with the aid of an entirely automatic apparatus which cuts the time for preparation of micrographic specimens of aluminum and all its light alloys extremely short.

FIRST PART

BIBLIOGRAPHY OF METALLOGRAPHIC APPLICATIONS OF ELECTROLYTIC

POLISHING OF ALUMINUM AND ITS ALLOYS

The references to all studies made from the end of 1947 to the end of 1950 are presented in the form of tables which contain the composition of the electrolyte, the particular application considered, the author, the title, and the bibliographic reference to the report.

The classification is based on the different types of electrolytes actually utilized. In this connection one will note that, with very few exceptions, all French reports have the polishing take place in perchloric acid - acetic-anhydride baths. In contrast, the foreign authors show a marked reluctance to make use of these mixtures and give preference to electrolytes where the perchloric acid is thoroughly diluted in an organic solvent (type of a De Sy-Haemers mixture) or absent. It is, in this case, replaced by nitric acid (type of the Waisman mixture), by phosphoric acid, or by fluoboric acid; these last two cases correspond to solutions used at the very beginning for polishing on an industrial scale.

The reputation of their dangerous character is the only cause for the fact that the mixtures of perchloric acid in the acetic anhydride have met with so little favor abroad, particularly in the Anglo-Saxon countries. This reputation dates from the spectacular explosion of an industrial tank at Los Angeles in 1947. Although the origin of that accident is not well known and later investigations have demonstrated the harmlessness of the acetic-perchloric mixtures used in the laboratories, of course on condition of a minimum of precaution, the distrust of a great number of metallographers could not be overcome. Thus, it came about that at English Universities perchloric-acid - acetic-anhydride baths are formally prohibited.

The nature of the polishing electrolytes may have a strong influence on the quality of the polish and, most of all, on the properties of the surfaces obtained. In fact, for certain types of mixtures, the polishing is accompanied by anodic oxidation. Furthermore, it is probable that the significance of this oxidation depends more or less on the conditions (temperature, density of the current) of the electrolysis. Thus it becomes difficult to predict a priori the chemical characteristics of the aluminum surface obtained. These characteristics do not apply, in general, for ordinary microscopy, that is, for the case where one is satisfied with observing the specimen under the optical microscope without subjecting it to other manipulations. On the other hand, special techniques, for instance that of etch figures, the obtainment of oxide replicas for electronic micrography (see p. 46), and especially the preparation of the test specimens destined for studies on plastic cold deformation, creep, fatigue, the electrochemical properties, etc., require the absence of any oxide film of notable thickness (larger than that of natural oxidation by air, that is, at the most of the order from 20 to 50 Å⁰).

In the actual state of the techniques for electrolytic polishing, the composition - perchloric acid (one part) and acetic anhydride (two parts) which corresponds to a standard anhydride medium - seems to be the only one which can give with simple precautions surfaces of aluminum on which the diffraction of electrons does not reveal the presence of alumina.

The misjudgment of the true chemical nature of the surfaces of aluminum polished electrolytically is likely to cause false interpretations of the results of certain studies undertaken on these surfaces. Regarding this matter, one will read with interest the study of G. R. Wilms (1) who confirms and specifies the cause of abnormal phenomena described by R. Maddin (2) - a cause that had already been suspected by several previous experimentators (discussion (3) of reference 2).

We shall have occasion to come back later on to these very important particulars (see p. 47). For the time being, one will have to keep in mind that any serious study involving surfaces of aluminum or of alloys

rich in aluminum, polished electrolytically, must necessarily contain an exact description of the conditions of this polishing and proof that the observations are not falsified by superficial impurities.

TABLES OF RECENT METALLOGRAPHIC APPLICATIONS OF ELECTROLYTIC

POLISHING OF ALUMINUM AND ITS ALLOYS

<u>Composition of the electrolyte</u>	<u>Particular application intended</u>	<u>References</u>
1. Electrolyte type perchloric acid		
A. Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of test specimens for study of the anisotropy created by abrasion striae	Farran, J.: Sur l'anisotropie optique superficielle des surfaces réfléchissantes striées, C.R., 224, 1947, p. 1,103.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of surfaces for study of the optic properties of films of aluminum obtained by by anodic oxidation	Huber, K., and Gaugler, A.: Über optische Untersuchungen an Deck-Schicht auf alumi- nium, Experimentia, 3, n° 7, 1947, p. 277.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Chemical anisotropy of crystals, plastic deformation, struc- tural imperfections	Lacombe, P., and Beaujard, L.: The appli- cation of etch figures on pure aluminum to the study of some micrographic problems, J. Inst. of Metals, 74, Sept. 1947, p. 1.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Micrography of aluminum crystals and of their boundaries	Lacombe, P., Beaujard, L., and Yannaquis, N.: Sub-boundary and boundary structures in high purity aluminum, Conference on Strength of solids, Bristol 1947.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Polishing arrangement for lim- iting the effects of etching on the surfaces	Farran, J.: Emploi d'une anode tournante dans le polissage électrolytique du Cd et de l'Al, Métaux et Corrosion, 23, janv. 1948, p. 9.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Intergranular dissolution of aluminum	Yannaquis, N., and Lacombe, P.: Influence de l'orientation des contours de grains de l'aluminium de haute pureté sur leur attaque par l'acide chlorhydrique, C.R., 226, 9 fév. 1949, p. 498.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Intergranular dissolution of aluminum	Lacombe, P., and Yannaquis, N.: La corro- sion intercrystalline de l'aluminium de haute pureté et ses conséquences au sujet de la nature des joints de grains, Rev. Métallurgie, 45, mars-avril 1948, p. 68; Métaux et Corrosion, 22, mars 1947, p. 35.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Differences between the bound- aries of grains and those of twin crystals from the view- point of fusion and of acidic attack	Chaudron, G., Lacombe, P., and Yannaquis, N.: Sur le comportement des joints de grains au cours du processus de fusion de l'aluminium très pur, C.R., 226, 26, avril 1948, p. 1,372.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of surfaces for study of films in case of anodic oxidation	Huber, K.: Pore volume of electrolytically produced protective coatings on aluminum, J. of Colloid Science, 3, juin 1948, p. 197.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Microscopic and X-ray study of the polygonization of alumi- num crystals	Guinier, A., and Lacombe, P.: L'état poly- gonisé du cristal métallique, Métaux et Corrosion, 23, sept. 1948, p. 212.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Structural differences between the films of anodic oxidation formed on aluminum and on zinc	Huber, K.: Structure et croissance des pellicules anodiques sur l'aluminium et le zinc, Colloque Intern. sur les réac- tions dans l'état solide, Paris, oct. 1948.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Micrography of Al-Zn alloys	Lacombe, P., and Berghezan, A.: Mise en évidence d'imperfections de structure de gros cristaux de solution solide Al-Zn au cours de leur durcissement structural, C.R., 226, 28, juin 1948, p. 2,152.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Effect of electrolytic polishing on the measurements of micro- hardness	Mouflard, M.: Etude critique des mesures de dureté et de microdureté, Métaux et Corro- sion, 23, nov. 1948, p. 245.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of surfaces for study of the diffraction of electrons of the metal and the films in natural oxidation	Raether, H.: La structure et l'oxydation des surfaces d'aluminium polies électrolytique- ment, C.R., 227, 1948, p. 1,247.

Composition of the electrolyte	Particular application intended	References
A. Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of test specimens for study of physical chemical processes (oxidation in air, corrosion by oils, action of hot gases)	Gwathmey, A. T., Leidheiser, H., Jr., and Smith, G. Pedro: NACA Tech. Note n° 1,460, juin 1948.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Crystallographic study of small isolated crystals	Lacombe, P., and Berghezan, A.: Sur l'existence de petits cristaux isolés dans les monocristaux métalliques de recristallisation et leur relation d'orientation, C.R., 228, 1949, p. 93. Metaux et Corrosion, 24, janv. 1949, p. 1.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Micrographic study	Lacombe, P., and Berghezan, A.: Influence de la répartition des imperfections de structure des cristaux de solution solide Al-Cu sur le mode de précipitation de la phase θ' , C.R., 229, 1949, p. 365.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Illustration of intragranular boundaries	Lacombe, P., and Berghezan, A.: Relation entre l'existence d'imperfections de structure des cristaux uniques d'aluminium pur et d'alliages Al-Zn et le processus de recristallisation, Physica, XV, avril 1949, p. 161.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Evolution of the micrographic structure in the course of hardening by tempering	Lacombe, P., and Berghezan, A.: Alcune recenti applicazioni della lucidatura elettrolitica all'esame micrografico delle soluzioni solide Al-Zn, e Al-Cu, Alluminio, 18, n° 4, 1949, p. 365.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	General report on the properties of electrolytically polished aluminum surfaces	Chaudron, G., and Lacombe, P.: New studies of the preparation of the surface of aluminum. Symposium on techniques of metallurgical examination, J. Inst. Metals, Birmingham, mars 1949.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Employment of thin and colored films of aluminum for the study of the structures of solidification, recrystallization, and deformation of aluminum	Lacombe, P., and Mouflard, M.: Les applications de la micrographie en couleurs à l'examen de l'aluminium et de ses alliages, Journées Métallurgiques de la S.F.M., 1949.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of test specimens for the study of the evolution of the superficial structure in the course of the fatigue of sheet metals in alternate torsion	Jacquesson, R., and Laurent, P.: Les renseignements relatifs à l'état cristallin des tôles fourmées par des essais de torsions alternées, Rev. Métallurgie, 46, fév. 1949, p. 89.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of surfaces before anodic oxidation	Huber, R.: Structure et croissance des pellicules anodiques sur Al et Zn, Bull. Soc. Chim. France, n° 3-4, 1949, p. D. 183.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparations of specimens before formation of the oxide layer serving as replica for micrographic study of precipitation by temperings, by means of the electron microscope	Castaing, R.: Recherches au microscope électronique sur les précipitations dans les alliages d'aluminium, C.R., 228, 1949, p. 1,341.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparations of specimens before formation of the oxide layer serving as replica for micrographic study of precipitation by temperings, by means of the electron microscope	Castaing, R., and Guinier, A.: Sur les images au microscope électronique des alliages Al-Cu durcis, C.R., 228, 1949, p. 2,033.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparations of specimens before formation of the oxide layer serving as replica for micrographic study of precipitation by temperings, by means of the electron microscope	Castaing, R., and Guinier, A.: Etude au microscope électronique du vieillissement des alliages Al-Mg-Si, C.R., 229, 1949, p. 1,146.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparations of specimens before formation of the oxide layer serving as replica for micrographic study of precipitation by temperings, by means of the electron microscope	Castaing, R.: Recherches au microscope électronique sur le vieillissement des alliages légers, Soc. Franc. Métallurgie, séance 21 dec. 1949.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Comparison of aluminum surfaces polished mechanically and electrolytically from the viewpoint of reaction with mercurous chloride	Bolognesi, G.: La reazione di superficie tra alluminio e cloruro mercurico, Alluminio, 18, sept.-oct. 1949, p. 493.

Composition of the electrolyte	Particular application intended	References
A. Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Test arrangement and results obtained in measuring - protected from air - the solution potential of aluminum polished electrolytically in the absence of air	Chaudron, G., Lacombe, P., and Youssouf, G.: Nouvelle détermination en l'absence d'oxygène du potentiel de dissolution de l'aluminium poli électrolytiquement, C.R., 229, 1949, p. 201.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Microscopic and X-ray study of structural imperfections of large crystals	Beaujard, L.: Nouvelles méthodes de préparation des cristaux d'Al et leurs applications à l'étude microscopique et cristallographique de l'état métallique, Publ. Sc. et Tech. du Ministère de l'Air n° 235, 1949, p. 57.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Micrography of the boundaries of polygonization	Lacombe, P.: Les différentes imperfections de structures des cristaux métalliques et leurs conséquences, Journées métallurgiques S.F.M. oct. 1949.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of test specimens for microscopic and X-ray study of the changes in surface structure of aluminum during creep	Wood, W. A., and Rachinger, W. A.: The mechanism of deformation in metals with special reference to creep, J. Inst. Metals, 76, 1949, p. 237.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of test specimens for microscopic and X-ray study of the changes in surface structure of aluminum during creep	Wilms, G. R., and Wood, W. A.: Mechanism of creep in metals, J. Inst. Metals, avril 1949, p. 643.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of test specimens for microscopic and X-ray study of the changes in surface structure of aluminum during creep	Wood, W. A., and Scrutton, R. F.: Mechanism of primary creep in metals, J. Inst. Metals, 77, 1950, p. 423.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Influence of electrolytic polishing on the speed of corrosion under stress	Perryman, E. C. W., and Hadden, S. E.: Stress - Corrosion of Al-Mg 7 percent alloy, J. Inst. Metals, 77, 1950, p. 207.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Influence of electrolytic polishing on the potential of the alloy under stress	Gilbert, P. T., and Hadden, S. E.: A theory of the mechanism of stress-corrosion in Al-Mg 7 percent alloy, J. Inst. Metals, 77, 1950, p. 237.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of the surfaces of aluminum alloys for replicas of anodic oxidation	Castaing, R.: Recherches au microscope électronique sur le vieillissement des alliages légers, La Recherche Aeronautique, n° 13, janv.-fév. 1950, p. 3.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of surfaces before the attack	Jacquesson, R., and Manec, J.: Figures de corrosion par attaque anodique, C.R., 230, 1950, p. 959. Journées d'Automne S.F.M. 1950.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of test specimens for fatigue tests	Jacquesson, R., Laurent, P., and Manec, J.: Remarques sur le comportement particulier d'un grain cristallin d'aluminium à la déformation mécanique, Métaux et Corrosion, 25, mars 1950, p. 61.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of test specimens for fatigue tests	Laurent, P. H.: Aspects particuliers de glissements produits dans des cristaux d'aluminium recuit, par des efforts alternés, Journées d'Automne S.F.M. 1950.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Micrographic examination of test specimens	Crussard, C., and Wyon, G.: Modification de structure de l'aluminium en cours de fluage, Journées d'Automne S.F.M. 1950.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Polishing of single crystal before deformation and of sections after deformation	Jacquesson, R., and Manec, J.: Etude métallographique de la formation d'une texture de laminage à partir d'un monocristal, Journées d'Automne S.F.M. 1950.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Preparation of surfaces subjected to the microhardness test	Roth-Meyer, H.: Etude de la microdureté des monocristaux au moyen du microscléromètre Girschig, Journées d'Automne S.F.M. 1950, C.R., 231, 1950, p. 906.
Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Study of surfaces with electron diffraction. Results not completely in agreement with those of H. Raether	Halfawy, H.: Sur le polissage électrolytique et l'oxydation anodique, Journées d'Automne S.F.M. 1950.

Composition of the electrolyte	Particular application intended	References
A. Perchloric acid - acetic anhydride (Jacquets' electrolyte)	Thinning of Al-Mg alloy specimens for taking X-ray diagrams (demonstration of internal stresses). Preparation of surfaces for micrography and for mercury attack	Jacquet, P. A., and Weill, A. R.: Sur la fissuration d'un alliage aluminium-magnésium au contact du mercure, Rev. de l'Aluminium, n° 172, déc. 1950, p. 442, n° 173, janv. 1951, p. 4.
ClO_4 (1 part) - acetic anhydride (6 parts)	Micrographic demonstration of the markings of plastic deformation. (After polishing, etch with: HF (4 parts) - NO_3H (1 part), glycerin (3 parts))	Banerjee, B. R.: A study of strain markings in aluminum, J. of Metals, sept. 1950, p. 1,126.
B. Perchloric acid - acetic acid (100 cm^3 $\text{CH}_3\text{CO}_2\text{H}$ - 30 cm^3 ClO_4H of 70-72 percent)	Preparation of test specimens for studies of corrosion and anodic oxidation	Gaugler, A.: Erfahrungen beim Glänzen von Al nach dem Verfahren, Comm. Privée, déc. 1947.
C. Perchloric acid (1 part), ethyl alcohol (4 parts)	Description of the technique of polishing for micrographic investigation of light alloys (with Cu, Cu-Ni, Mg-Zn-Mn, Gl, etc.)	Walton, M. B., Middleton, B. F., and Mott, B. W.: Electrolytic polishing of aluminum alloys, R.A.E. Report n° M.E.T. 9, juin 1946.
Perchloric acid (1 part), ethyl alcohol (4 parts)	Description of the polishing method applicable to all aluminum alloys except those containing more than 2-percent silicon	De Sy, A. L., and Haemers: Rapid electro-polish and etch, Metal Progress, 53, mars 1948, p. 368.
Perchloric acid (1 part), ethyl alcohol (4 parts)	Micrographic investigation by electrolytic polishing and etch figures on aluminum	Bowles, J. S., and Boas, W.: The effect of crystal arrangement on "secondary" recrystallization in metals, J. Inst. Metals, 74, 1948, p. 501.
Perchloric acid (1 part), ethyl alcohol (4 parts)	Polishing of aluminum in order to take replicas of rough surfaces which have afterwards been anodically oxidized	Nutting, J., and Cosslett, V. E.: A replica technique for the examination of fracture surface with the electron microscope, Symposium in Metallurgical applications of the electron microscope, Londres, nov, 1949.
Perchloric acid (1 part), ethyl alcohol (4 parts)	Micrographic determination of the solubility of tin in aluminum	Sully, A. H., Hardy, H. K., and Heal T. J.: The aluminum - tin phase diagram and the characteristics of Al alloys containing tin as an alloying element, J. Inst. Metals, nov. 1949, p. 269.
Perchloric acid (1 part), ethyl alcohol (4 parts)	Micrographic examination of aluminum containing slight proportions of another element (Ag, Gl, Cr, Cu, Fe, Mg, Mn, Ni, Si, Ti, Zr)	Harrington, R. H.: The effect of single additions metals on the recrystallization, electrical conductivity and rupture strength of pure aluminum, Trans. Amer. Soc. Metals, 41, 1949, p. 443.
Perchloric acid (1 part), ethyl alcohol (5 parts)	Study - by means of electron microscope - of the slip lines formed on test specimens of ultra-purity aluminum deformed between -180° and $+500^\circ$ C	Brown, A. F.: Elementary slip processes in aluminum as shown by the electron microscope, Symposium on metallurgical applications of the electron microscope, Londres, nov. 1949. Brown, A. F.: Fine structure of slip zones, Nature, 163, 18 juin 1949, p. 961.
60-percent perchloric acid (50 cm^3) - alcohol (200 cm^3)	Micrographic examination of specimens of aged solid solution Al-Zn	Perryman, E. C. W., and Blade, J. C.: Relationship between the aging and stress-corrosion properties of Al-Zn alloys, J. Inst. Metals, mai 1950, p. 263.
D. Perchloric acid (15 cm^3) - methyl alcohol (85 cm^3)	Study of polished surfaces, with electronic diffraction (aluminum diagram)	Raether, H.: La structure des surfaces d'aluminium polies électrolytiquement et leur oxydation, Metaux et Corrosion, 25, janv. 1950, p. 1.
60-percent perchloric acid (2 parts), methyl alcohol (8 parts)	Preparation of surfaces for oxide replicas intended for examination by means of electron microscope	Communication Privée de Geach (Research Laboratory of Associated Electrical Industries - Ltd, Angleterre). Voir aussi G. L. Bucknell and G. A. Geach, Nature, 164, 6 août 1949, p. 231.
60-percent perchloric acid (2 parts), methyl alcohol (8 parts)	Electronic micrographs of the aging of Al-Cu alloys	Bucknell, G. L., and Geach, G. A.: Observation on the age hardening process in an aluminum-copper alloy, Symposium on metallurgical applications of the electron microscope, Londres, nov. 1949.

Composition of the electrolyte	Particular application intended	References
II. Electrolytes of phosphoric acid type		
A. Phosphoric acid - ethyl alcohol (400 PO_4H_3 , d = 1.75, 380 alcohol - 200 water)	Polishing of aluminum and light alloys rich in Ag for micrographic examination. The method causes production of an aluminum film	Larke, L. W., and Wicks, E. B.: A method for the electrolytic polishing and etching of some Al-Ag alloys commercial purity aluminum and pure magnesium, <i>Metallurgia</i> 41, janv. 1950, p. 172. R.A.E. Technical Note n° M.E.T. 106.
Phosphoric acid - ethyl alcohol (400 PO_4H_3 , d = 1.75, 380 alcohol - 200 water)	Surface preparation of fatigue-test specimens in Al-Mg alloys for studying the evolution of the micrographic structure	Forsyth, P. J. E.: Some metallographic observations relating to the fatigue of metals, R.A.E. Report M.E.T. 54, juin 1950.
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B. Phosphoric acid - carbitol (monoethyl ether of diethylamino ethanol)	Micrographic examination of hot or cold rolled aluminum subjected to creep	Méthode utilisée au National Physical Laboratory par D. McLean et ses collaborateurs (<i>Nature</i> , 165, 14 janv. 1950, p. 70); (<i>Journées d'Automne S.F.M.</i> 1950.)
Phosphoric acid - carbitol - hydrofluoric acid (exact composition not indicated)	Micrographic detection of the G phase and of Mn Al_6 in the 2.52-percent Mn alloy	Marchand, G.: Discussion du mémoire de K. Little, G. V. Raynor, and W. Hume-Rothery, (<i>J. Inst. Metals</i> , 1947, p. 83), <i>J. Inst. Metals</i> , août 1947, p. 747.
(85-percent PO_4H_3 70 percent - carbitol 26.5 percent - H_2O 2.5 percent - FH 1 percent)	Surface preparation for the production of aluminum film serving for determination of orientations. Oxidation is carried out in the same bath, under conditions different from the polishing conditions	Hone, A., and Pearson, E. C.: Grain orientation in aluminum revealed by anodic film, <i>Metals Progress</i> , 53, mars 1948, p. 363.
(85-percent PO_4H_3 53 percent - carbitol 20 percent - H_2O 26 percent - FH 1 percent)	Micrographic study of the growth of grains by means of anodic-oxidation films obtained in the polishing bath under changed conditions	Sperry, P. R.: Method for studying grain boundary migration of aluminum, <i>J. of Metals</i> , janv. 1950, p. 103.
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(85-percent PO_4H_3 53 percent - carbitol 20 percent - H_2O 26 percent - FH 1 percent)	Micrographic study of the growth of grains by means of anodic-oxidation films obtained in the polishing bath under changed conditions	Beck, P. A., and Sperry, P. R.: Strain induced grain boundary migration in high purity aluminum, <i>J. Applied Physics</i> , 21, fév. 1950, p. 150.
Distilled water 1,012 cm^3 - Oxalic acid 72g - Anhydrous boric acid 128g - 85-percent orthophosphoric acid 808 cm^3 - carbitol 1,660 cm^3 - 48-percent fluoric acid 320 cm^3	Polishing and anodic oxidation for studying the shape of grains, the plastic deformation, and the recrystallization of aluminum	Hone, A., and Pearson, E. C.: A new anodic film method for studying orientation in aluminum, <i>Metal Progress</i> , 58, nov. 1950, p. 713.
C. Phosphoric acid - sulphuric acid - (PO_4H_3 15 percent - H_2SO_4 70 percent - water 15 percent)	Micrography of commercial aluminum	Herenguel, J.: Généralités sur les textures d'orientation préférentielles, <i>Rev. Métallurgie</i> , 45, dec. 1948, p. 505.
Phosphoric acid - sulphuric acid - (PO_4H_3 15 percent - H_2SO_4 70 percent - water 15 percent)	Micrography of commercial aluminum	Herenguel, J.: Analyse d'une texture de solidification du type basaltique, <i>Rev. Métallurgie</i> , 46, mai 1949, p. 309.

<u>Composition of the electrolyte</u>	<u>Particular application intended</u>	<u>References</u>
C. Phosphoric acid - sulphuric acid - (PO ₄ H ₃ 15 percent - H ₂ SO ₄ 70 percent - water 15 percent)	Micrography of Al-Mg alloys	Herenguel, J., and Segond, R.: Emploi de l'oxydation anodique pour l'étude de l'homogénéité des solutions solides Al-Mg, Rev. Metallurgie, 46, juin 1949, p. 377.
Phosphoric acid - sulphuric acid - (PO ₄ H ₃ 15 percent - H ₂ SO ₄ 70 percent - water 15 percent)	Micrography for perfecting light alloys susceptible to electrolytic polishing	Herenguel, J., and Segond, R.: Alliages légers spéciaux pour le polissage électrolytique, Rev. Aluminium, n° 148, oct. 1948, p. 306.
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Phosphoric acid - sulphuric acid - (PO ₄ H ₃ 15 percent - H ₂ SO ₄ 70 percent - water 15 percent)	Micrography of 99.5-percent cast aluminum	Herenguel, J.: Etude micrographique des textures de solidification de l'aluminium de titre 99.5 percent (Texture d'hétérogénéité), Rev. Metallurgie, 47, janv. 1950, p. 29.
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<u>Composition of the electrolyte</u>	<u>Particular application intended</u>	<u>References</u>
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SECOND PART

DESCRIPTION OF SEVERAL MICROGRAPHIC TECHNIQUES

USING ELECTROLYTIC POLISHING

I. Recent Polishing Methods

The majority of experimentators who use electrolytic polishing for their metallographic investigations (microscopy, preparation of test specimens, etc.) do not give any information on operating conditions; sometimes they do not even indicate the composition of the bath.

As the bibliography, which was presented in the preceding tables, shows, the electrolytes for polishing of aluminum comprise several types, and even for the same type the proportions of the constituents may vary around certain values.

Since publication of our report on electrolytic polishing of aluminum and its alloys, no new methods have been made public, at least in detailed form which would justify an analysis.

To our knowledge, only two investigations undertaken at the Royal Aircraft Establishment at Farnborough deserve to be described. The older one (4) concerns a method utilizing a perchloric-acid - alcohol electrolyte derived from that of De Sy and Haemer. The second (5) has the special object of micrographic polishing of aluminum-silver alloys rich in silver for which the customary techniques do not yield good results. Besides, it has been found that the new procedure may be applied with equal success to other alloys like magnesium alloys.

(a) Perchloric-acid - alcohol electrolyte.- A first investigation of the R.A.E. (6) has shown that Jacquets'¹ method is applicable to certain aluminum alloys. It had also been determined that the optimum polishing is obtained when the anode is in horizontal position. The authors indicated, furthermore, that the surfaces subjected to electrolysis should be polished mechanically on a velvet impregnated with a polishing composition.

A surface prepared with emery 000 after electrolysis would present an exaggerated relief of the intermetallic constituents. According to our experience, these observations probably do not apply to polishing in the more concentrated acetic-perchloric solution which we perfected in 1943 and of which the R.A.E. had not been cognizant.

Since it had been established on the other hand that according to De Sy and Haemers the perchloric-acid - alcohol electrolytes achieve an excellent polishing of light alloys in very short times, Waldron, Middleton, and Mott planned to study this type of bath more thoroughly.

First of all they determined, by means of the conventional potentiometer technique, the current-voltage curve for different compositions of the bath and values of the anodic surface (anode constituted to the alloy RR 56). In order to trace this curve, a rapid cooling-off of the electrolyte is indispensable (by means of a copper tube through which a stream of water flows functioning as cathode). The intensity of the current shown on the diagram is the value at equilibrium if the limiting potential is gradually increased by small amounts between 0 and 20 volts. In all cases, the curve obtained is typical for electrolytic polishing, that is, it shows a maximum intensity in general, between 4 and 8 volts. The beginning of this stage coincides with the removal of a black film

¹This concerns the original method (7), not the improved technique described later (8).

which appears first on the anode at low values of voltage.² The best polishing is obtained toward the end of the stage which is also in accord with the conventional cases. Under lower voltage, the constituents are less well defined whereas for higher values the surface is clearly defined and a preferential attack appears around the constituents.

As we said before, the respective proportions of the perchloric acid and the alcohol do not have a very great influence on the shape of the current-voltage curve. The composition is of the type: four parts 74.5-percent ethyl alcohol and one part 60-percent perchloric acid, in the case of a titrating 12-percent ClO_4H mixture. However, with a more diluted electrolyte (for instance 9-percent ClO_4H) the drop in intensity before the stage is less easily detectable; correlatively, the polishing is less satisfactory (preferential attack around the constituents).

Several types of alloys have been tested. The initial state of the surface is the rough polish accomplished by means of abrasive papers up to and including 000. The results vary according to the composition of the intermetallic phases. A solid solution can always be polished perfectly, but with insoluble constituents avoidance of every relief effect is impossible. However, this relief is frequently less notable than that obtained by normal mechanical polishing, and the distinctness of the particles is not affected by it.

One also notes that for certain alloys a film is produced which impedes microscopic investigation. This film may generally be eliminated by immediate rinsing of the polished surface in a violent hot-water jet followed by very light mechanical polishing on a cloth moistened with an aqueous suspension of fine alumina.

Aluminum-silicon alloys: Electrolytic polishing is unsatisfactory because the silicon particles do not dissolve and remain in relief imbedded in the matrix.

Nevertheless, in case of alloys containing silicon in the form of a slightly modified fine eutectic the relief effect is not sufficiently pronounced to be detrimental to an investigation if that investigation has the purpose of observing the degree of modification of the eutectic. The electrolytic procedure may then be utilized as a rapid control method.

Duraluminum-type alloys: The surface obtained is not very good for examination of the general structure under slight magnifications. On the other hand, the constituents do not appear sufficiently distinct under large magnifications.

²Elimination of this black film and also of the small gas bubbles which tend to adhere is attained by gently agitating the specimens. A mechanical stirring of the bath is effective but perturbs the electric conditions and, consequently, does not permit a correct tracing of the curve.

For alloys of this type one will doubtlessly have to resort to another method.

Copper and copper-nickel alloys: The alloys of the English specifications D.T.D. 304 and B.S.S.L. 40 undergo successfully electrolytic polishing for micrography. At a magnification 500, the particles of the constituent Al-Cu-Fe in the alloy D.T.D. 304 thus are very distinct without relief effect.

"High-resistance" type alloys: The alloys Al-Mg-Mn-Zn in the heat-treated state are easily polishable. The intermetallic constituent which sometimes appears in this type of alloy is very manifest so that electrolytic polishing has here been utilized with success for a systematic study of the structure.

In an experimental alloy of the same type containing, in addition, chromium, there exists an unidentified phase susceptible to attack which appears in the form of black circles after prolonged polishing. This phase is added to the normal constituent rich in manganese and in chromium.³

Light alloys containing a highly dispersed constituent: The methods of electrolytic polishing have proved to be extremely useful for study of the structure of alloys where the slight hardness or brittleness makes mechanical polishing difficult. These special characteristics may result from separate or simultaneous effects of the composition or of thermal treatment.

Thus it is possible to study the very fine structure of the eutectic Al-Be containing only 0.6 percent of beryllium which is extremely likely to creep on the surface in case of mechanical polishing. After electrolytic polishing, the eutectic appears in the form of a soluble aggregate in which the primary beryllium particles show a brownish tint.

Electrolytic polishing has been employed systematically for a study of the mode of precipitation of the β phase of a 5-percent Al-Mg alloy which had been subjected to various mechanical and thermal treatments. The solid solution takes an excellent polish and the intermetallic phase is well defined, without any relief.

An attack of 45 min in 9-percent orthophosphoric acid has revealed precipitation in the interior and in the grain boundaries. Only an extremely careful mechanical polishing could give results equivalent to those of electrolytic polishing which is incomparably faster and easier.

³The original report contains a micrograph at magnification 500 which shows the presence of this intermetallic constituent perfectly analogous to those we had observed in the French alloys zicral and T 60 (see p. 44).

Conclusion: In summary one may say that an electrolyte consisting of perchloric acid (approximately one part) and ethyl alcohol (approximately four parts) is suitable for the polishing of several types of aluminum alloys. One is led to suppose that the attack of the intermetallic constituents depends to a great deal on the viscosity of the solution and its content of hydrogen ions.⁴

According to the authors, the polishing is completed in one or two minutes, that is, in a considerably shorter time than with Jacquets' method (we do not agree on this particular point). On the other hand, the mixture does not have an unpleasant odor, can be easily prepared, and does not have any explosive characteristic.

The employment of electrolytic polishing in metallography of light alloys appears limited by various considerations. There exists little probability that one single solution could be suitable for all light alloys. Each case will require the perfecting, or possibly the discovery of an electrolyte and of the most expedient technique. Thus it will be necessary for a laboratory to possess the equipment capable of undergoing the desired modifications.

Incidentally, we want the reader to note that this last opinion presented by the English authors in 1946 has been contradicted by later research. Actually, as we shall see, it is now perfectly feasible to satisfactorily polish all light alloys of technical interest, that is, alloys with an aluminum base, in all their states, by means of a unique technique and with the utmost rapidity.

(b) Phosphoric-acid - alcohol electrolyte.- For study of the solubility limit of silver in aluminum, the methods suited best are micro-graphic examination and measurement of the electric resistance.

The first method runs into great difficulties due to the very slight hardness of the alloy. Attack by conventional reagent (diluted hydro-fluoric acid, for instance) produces effects of etching which render the distinctness of the precipitate in the solid solution uncertain. In fact, it has not been possible to perfect a purely mechanical technique, and the experiments have led to a procedure of electrolytic polishings.

None of the known electrolytes produces entirely satisfactory results. The perchloric-acid - alcohol mixture described above is suitable only for an alloy of very low silver content. Finally, the optimum conditions found are the following:

⁴The viscosity certainly plays a large role. This is doubtlessly the reason for the fact that the addition of glycerin or of butylcellosolve greatly improves the polishing of heterogeneous alloys in alcoholic solutions of perchloric acid.

The electrolyte consists of 400 cm³ phosphoric acid (density 1.75), 380 cm³ absolute ethyl alcohol (density 1.30), and 200 cm³ distilled water. The density of this mixture is 1.25.

The cell is a beaker of 1,000 cm³ containing a semicircular aluminum cathode. The specimen (a parallelepiped 2 cm long and with a section of 1 cm², total surface = 10 cm²) is pierced in its lower part by an aperture into which one forces an aluminum wire which is connected with the positive pole. A mechanical glass stirrer is placed at the center of the cell between the two electrodes. The limiting voltage is applied by means of resistances placed in series in the circuit.

The state of the initial surface corresponds to rough polishing by abrasive papers through 000 followed by rapid rubbing on velvet coated with commercial polishing paste. This preliminary preparation must be followed by a degreasing by means of benzine.

When a positive connection with the wire has been made, the assembly is immediately immersed in alcohol before its introduction into the cell. The surface to be polished is horizontal, parallel to the surface of the liquid and at a distance of several millimeters from the latter. The stirrer rotates at approximately 400 rpm.

The optimum electric conditions vary slightly with the age of the bath and the temperature.

In a freshly prepared bath, maintained at a temperature between 42° and 45° C, the density of the current is of the order of 35 a/dm² with a voltage of 27 to 30 v. Under these conditions, the polishing is completed in 4 to 6 minutes.

A current density lower than 35 a/dm² or a temperature below 40° C entails the formation of an anodic film on all Al-Ag alloys. However, if the intensity exceeds 35 a/dm² or if the temperature rises above 45° C, the surface is clearly defined.⁵

If the bath is enriched in aluminum phosphate, its electric resistance varies which has the effect of enlarging the optimum temperature range for the same density of current but with a voltage of under 35 v.

⁵These observations tend to indicate that the mechanism of polishing implies the formation and dissolution of an aluminum film. The same phenomenon is produced with phosphochromic electrolytes. The possibility of an anodic oxidation subsequent to the polishing effect is an unfavorable factor in applications of these baths for purposes other than ordinary micrography (see p. 47).

Evaporation of the electrolyte is eventually compensated for by addition of a desired quantity of a mixture of four parts alcohol and two parts distilled water, in order to re-establish the same value of density.

Results: The alloy in the state of solid solution exhibits to micrographic investigation a very well polished surface on which only the grain boundaries stand out, the definition of which is a function of the silver content. Intensification of this attack may be obtained by making the surface emerge above the bath (the attack then results in a heating of the liquid film).

At the end of polishing and attack, the specimen is withdrawn and immediately immersed in hot water in order to prevent stains. The solid phosphates possibly deposited are removed by gentle rubbing with a cotton tampon soaked in alcohol. One then washes the specimen in alcohol and dries it by means of a lukewarm air jet.

This method gives excellent results with all Al-Ag alloys examined (2.4 and 8 percent Ag), for different structural states. However, each case may require slight modifications of the conditions.

Applications to other light materials: Under the same conditions of current density and of temperature, the aluminum of commercial purity (99.5 percent) also can be polished very well (the micrograph here presented shows, for instance, in an extremely well-defined form, a nodule of the ternary eutectic $\text{Al-Al}_4\text{FeSi}_2\text{-Si}$).

The technique may be applied equally well to pure magnesium; however, the cathode then is of magnesium, and the density of the current 20 a/dm^2 under a reduced voltage of 10 v. The temperature control is less critical. At the end of the electrolysis, the anode is withdrawn under current, immediately immersed in cold water, then in alcohol, and dried by means of a cold-air jet. The film which is sometimes visible on the surface when it is withdrawn from the alcohol is removed with cotton soaked in concentrated nitric acid.

We remark that, according to our observations published a long time ago (9), the magnesium may be polished in another phosphoric-alcohol mixture and under rather different conditions of current density and of temperature. However, the method described above offers the great advantage of greater rapidity, while the micrographs published show nevertheless an excellent quality of polishing.

II. Chemical Polishing

Obtaining smooth and shiny metallic surfaces by simple immersion in acid solutions, without passage of an electric current, is now possible for certain metallic materials, including in particular, aluminum and some of its light alloys. This process, known by the name of "superpolish" ("brillantage") or chemical polishing, has been discovered more or less independently in several laboratories one of which is the Battelle Memorial Institute, well known for its research concerning electrolytic polishing on an industrial scale. Until now, this method has been intended only for the finishing of fabricated objects but it might possibly be of a certain advantage in micrography although it would continue to be applied - at least in its present form - much less generally than electrolytic polishing.

It is with regard to this eventual development, predicted recently by J. Herenguel (10) that we shall give a few hints concerning the chemical polishing of aluminum. In the technical literature (11), one will find reports dealing with the method in general.

"Superpolish" of magnesium.- It has been known for a long time that magnesium may be made brilliant by immersion in a concentrated mineral acid. We have put this property to use for eliminating, before the electrolytic polishing, the surface layers disturbed by mechanical preparation (9).

In his investigations on the protection of magnesium by anodic oxidation in an alkaline medium at the Office National d'Etudes et de Recherches Aéronautiques (O.N.E.R.A.), P. Galmiche used a concentrated alcoholic solution of hydrochloric acid for shining the surface before treatment. This same procedure has been rediscovered quite recently in the Laboratory of LeBrocq at Farnborough (25).

No investigation has been made on the possible application of chemical "superpolish" in the micrography of magnesium and of magnesium-aluminum or magnesium-manganese alloys.

"Superpolish" of aluminum.- In the Battelle method, solutions are used which contain in different concentrations the three acids: phosphoric, nitric, and acetic acid. The composition of the baths recommended by Aluminum Français (Alupol method) is not specified. One of these baths produces a simple luster while another one has, on the other hand, a very distinct polishing effect.

Here follow some data on the method studied by the Society and Enthone Company of America. Experience there has shown that mixtures of phosphoric acid, nitric acid, and water make aluminum and its alloys brilliant if they are immersed for 15 seconds to 10 minutes in the solution heated to between 98° and 138°. The fact that certain mixtures have an effect of polishing

and shining while others achieve only a mat finish seems to be connected with rapidity of dissolution of the metal. This velocity is highest in the phosphoric acid, decreases by addition of acetic acid, and even more in the presence of the third acid, the nitric acid. Table I indicates the appearance of the surface obtained with these four types of solutions.

TABLE I

Materials	H_3PO_4 -Water	H_3PO_4 - $\text{CH}_3\text{CO}_2\text{H}$ - H_2O	H_3PO_4 - HNO_3 - H_2O	H_3PO_4 - $\text{CH}_3\text{CO}_2\text{H}$ - HNO_3 - H_2O
2-1/2 H	Shining and slight polishing in 3 minutes	Slight shining in 3 minutes	Shining and polishing in 3 minutes	Shining and polishing in 3 minutes
24T3	Etching in 3 minutes	Shining in 15 sec and etching in 3 minutes	Shining in 30 sec. Attack producing a milky surface	Shining, then polishing in 3 minutes
52-1/2 H	Etching in 3 minutes	Shining in 15 sec and attack in 3 minutes	Shining, then polishing in 3 minutes	Shining, then polishing in 3 minutes

2-1/2 H is 99-percent aluminum cold worked.

24T3 is the alloy with 4.5-percent Cu - 1.5-percent Mg and 0.6-percent Mn heat treated.

52-1/2 H is the alloy with 2.5-percent Mg and 0.25-percent Cr cold worked.

The mixture finally left contains phosphoric and nitric acid and is used at a high temperature which poses problems of tank maintenance, vapor elimination, and modification of the composition. The exact temperature and the duration of the immersion vary with the character of the light alloy and its structural state. Each case therefore requires a special perfection of method which must be made on the object itself since the degree of polish obtained depends on the initial state of the surface brought about by the mechanical operations of shaping (embossing, stamping, rolling, etc.).

The baths studied by the Tréfileries and Laminaires of Le Havre contain phosphoric, sulfuric, and nitric acid. The polishing is done in two stages. The first, with a solution rich in sulfuric acid provokes a violent attack. The second, more selective, takes place in a concentrated phosphoric-acid mixture.

For micrographic applications, Herenguel recommended the solution PO_4H_3 (70 percent) - H_2SO_4 (25 percent) - HNO_3 (5 percent).

The future use of chemical polishing in the domain of micrography is certainly very limited. This method may give industrially interesting results with aluminum and its homogeneous alloys, but the quality of polish required for the study of structures, even the simplest ones, is perhaps beyond its possibilities. In any case, the operation will require precise control of several factors, much harder to realize than with the present methods of electrolytic polishing.

III. Anodic Oxidation

The employment of anodic oxidation as micrographic reagent for aluminum and its alloys we owe to Lacombe. Since this surface state is indispensable for the preliminary preparation of the specimens, the principle of the method and its first results have been described in our report on electrolytic polishing.

Lacombe and his coworkers have demonstrated the advantages of anodic oxidation for revealing minute structural details in very high-purity aluminum (cold working, recrystallization, etc.) and the traces of chemical heterogeneities of certain solid solutions. The technique leads to micrographs in particularly spectacular colors.

In consequence of this work, other authors have confirmed the advantages of anodic oxidation, for instance, for following the evolution during the time taken by the phenomena of recrystallization and of growth of grains. From this point of view, the studies of Beck and his coworkers at the American University of Notre Dame deserve to be pointed out (12).

The method used for revealing the grain-boundary migration in the course of annealing of very high-purity aluminum consists in making each heating period be followed by an anodic oxidation. The preceding traces are still visible, and the actual grain is detected by the color of its film of oxide examined in polarized light. There follow a few operative details.

The specimen containing grains of the desired shape is slightly deformed by rolling or compression, then polished electrolytically with the automatic apparatus Buehler-Waisman in an electrolyte containing a volume of 53 percent of 85-percent orthophosphoric acid, 26 percent of distilled water, 20 percent of carbitol (monoethylether of diethylene-glycol), and 1 percent of 48-percent hydrofluoric acid. The following anodic oxidation takes place in the same bath but under a weaker voltage and density of current. The duration is between 1 minute 30 seconds and 2 minutes.

Thus the structure of aluminum is revealed by three effects.

- (a) A selective attack of the boundaries.
- (b) A heterogeneous attack on the surface of the cold-worked grains.
- (c) Finally, the coloration in polarized light which is characteristic for the orientation of every crystal.

After having photographed a chosen point of the surface thus prepared, one follows up with a short annealing, for instance, 1 minute at 400°C ; then the film of oxide resulting from the first attack is eliminated by an immersion of 1 to 2 minutes in a phosphochromic mixture (35 cm^3 of 85-percent PO_4H_3 and 20g of CrO_3 per liter), heated to 100°C .

A new anodic oxidation shows by the color in polarized light the new form of the grain considered, with the previous traces still visible owing to the selective attack of the grain boundaries. The sequence of the processes: annealing dissolution of aluminum oxidation may be repeated as often as desired.

In these experiments, one can see that the grain increases in size at the expense of the neighboring grain still cold worked, for on one side the surface is very smooth, whereas on the other it presents the characteristic etching of a deformed network. Besides, an X-ray examination confirms these two structural states. Thus the process corresponds to a recrystallization without formation of new nuclei.

Beck and his coworkers then attempted to specify the relations of mutual orientations which favor the growth of a grain at the expense of its neighbor. Under the assumption that a spontaneous formation of recrystallization nuclei is rather rare even in pure aluminum cold worked to 40 to 50 percent by rolling, the authors had to develop what they call an artificial nucleation. For this purpose, the metal deformed to 12 percent, is electrically polished; then, a fine ruling is traced on its surface which induces a supplementary cold working. After that, one performs an annealing of 1 hour at 350°C , then a short electrolytic polishing for partly erasing the ruling, and finally an anodic oxidation.

On the trace of the ruling and in its immediate neighborhood, several small grains of recrystallization have formed and certain grains grow at the expense of the matrix deformed to 12 percent. One now determines by means of the technique of etch figures and the goniometer the relative orientations of the grain in the course of growth and its cold-worked neighbor at whose expense this growth takes place, and finds that that of the recrystallized grain corresponds to a rotation of about 40° of the planes (III) of the other.

On the other hand:

- (1) A grain never grows at the expense of a cold-worked grain of almost the same orientation.

(2) The displacement of the boundary between two grains in twin-crystal position (rotation of 0° or 60°) of the planes (III) is very slight.

This example shows the advantage of the technique of anodic oxidation for studying with the microscope the phenomena of recrystallization and of growth of aluminum crystals. The method is probably generalizable for metals which can, like aluminum, be covered by thin oxide films by electrolytic process; this is the case, for instance, for uranium (26) and titanium (27) for which a method of electrolytic polishing also is known.

IV. Etch Figures

The employment of the etch figures has been known for a long time to mineralogists as a rapid means of determination of the crystallographic orientations. Metallurgists were equally interested in it, ever since the first beginnings of micrography; however, at that stage, the polishing technique for specimens did not permit attainment of the hoped for results because - as we now know - the abrasion and the mechanical polishing produce a superficial cold-worked layer which conceals the true crystal-line structure.

With electrolytic polishing, this difficulty disappeared; it was Lacombe who as the first thought of profiting from the state of surface thus produced for developing the applications of the method of etch figures in the case of pure aluminum. The experimental details are well known now, likewise the important results already found (13). Let us remember among them only the evidence of the boundaries of polygonization and of the heterogeneities of concentrations of solid solutions (28).

The conventional technique of Lacombe, that is, the attack with a mixture of the three acids, nitric, hydrochloric, and hydrofluoric acid, may be applied satisfactorily only to crystallized or slightly deformed aluminum. For cold-worked metal, Jacquesson (14) has shown etch figures by anodic attack which, according to him, yields the best results.

The anode is formed by the specimen itself in a solution of diluted hydrochloric acid. The only factors are the density of the current and the duration of the electrolysis while the concentration of the acid plays only a secondary role.

The duration of attack is of the order of 5 sec. When the density of the current is lower than 0.5 a/cm^2 , the etch figures are not numerous but very large, and their contour is irregular and very variable. They are the less serviceable, the lower the density of the current.

For densities of the order of 0.8 a/cm^2 , or more, the figures are very small (a few microns) and extremely numerous. Their geometrical form is simple. They can be used only at high enlargements but they give the orientation of very small crystals and thus offer great advantage in the study of highly irregular textures.

For aluminum, the most favorable intensity is 0.6 a/cm^2 . The figures have clear, geometrically simple outlines, an approximately constant shape, and are regularly distributed over the surface. They measure 2 to 3 mm at 700-x enlargement and permit very good determinations of orientation. Finally, they maintain their distinctness whatever the amount of cold working.

It is important to remark that the presence of an alumina film greatly disturbs the uniformity of the anodic attack; the latter requires an electrolytic polishing in an acetic-perchloric bath in good condition. When the conductivity of the bath becomes too great, a detrimental superficial oxidation is produced.

The effect of an addition of nitric acid or of hydrofluoric acid to the hydrochloric acid is a lowering of the optimum density of current. The clearness of the etch figures is increased and, above all, they take on greater depth.

In a mixture 5-percent HCl, 2-percent HNO_3 , and with 0.6 a/dm^2 the pits are very numerous; they seem to form preferably on the grain boundaries and the cold-working residues which appear on the surface as black lines.

This method of producing etch figures is characterized by its flexibility. It is possible to obtain data on one or the other particulars of the crystalline texture by merely modifying the amount of density of the current.

Attack on the grain boundaries.- It is known that in the metallography of aluminum and its alloys it is sometimes difficult to reveal the grain boundaries with the customary reagents, even when the surface is electrolytically polished. This difficulty arises either in the case of aluminum, the crystals of which have a preferred orientation, or in that of the alloys without intergranular heterogeneity. This last case will be treated later on. (See p. 42.)

Although one cannot, properly, speak of an attack by etch figures, we shall summarize here the technique described by Herenguel and Santini (15) for making the grain boundaries in aluminum grains of preferred orientation visible.

The method affects selectively the boundaries, that is, it does not notably etch the grains themselves. It consists in placing the electrolytically polished test specimen as cathode in a solution containing in volume:

40-percent hydrofluoric acid	10 percent
Pure glycerin	55 percent
Distilled water	35 percent

This electrolyte functions at ordinary temperature. The density of the current is about 1.5 a/dm² (voltage 7 to 8 v) with a duration of 5 minutes. The surface was rapidly rinsed after being taken from the bath.

The method has been used for 99.5-percent aluminum. The authors do not specify whether it is applicable also to the high-purity metal or to light alloys.

Observation of the micrographic structures of deformation.- For copper and its alloys, it is relatively easy to show by micrography the beginning of plastic deformation (16). The problem seems more difficult for aluminum, probably because a sufficiently sensitive reagent is lacking.

In a recent report, B. B. Banerjee studied this problem and showed that very high-purity aluminum (99.998 percent) must have undergone a reduction in thickness of at least 12 percent, to permit observation by microscope to detect clear signs of cold working (17).

The technique consists in deforming single crystals either by compression with a press, or by impact, or by rolling, using a specimen of a special form so as to obtain a deformation gradient varying from 0 to 50 percent over the length.

The deformed specimen is polished mechanically, then electrolytically. The two best polishing methods use:

1. 12 parts of concentrated nitric acid, 56 parts of methyl alcohol, 3 parts of chromic acid (in saturated solution), 8 parts of acetic acid, and density of current of about 100 a/dm² and 42 volts.

2. Perchloric acid 1 part - acetic acid 6 parts - density of current very weak, under 45 v.⁶

⁶Elsewhere in his report, the author indicates a bath of perchloric acid and acetic anhydride. We suppose that he is dealing with the same mixture, as Americans do not always make a distinction between acid and anhydride in their publications.

Banerjee declares, after having tested several electrolytic reagents that he obtained the best results with the mixture: hydrofluoric acid 4 parts - nitric acid 1 part - glycerin 3 parts.

The memorandum is open to some criticism. Thus the author insists on the necessity of avoiding any heating up during the mechanical polishing which follows the deformation, so as not to cause a temperature effect on the cold-worked structure. However, he does not take the same precaution in the course of the electrolytic polishing (which would in addition be difficult). Particularly the bath which functions under 100 amperes per cm^2 produces with certainty a relatively considerable rise in temperature. We are of the opinion that this work requires more serious consideration. It should be possible, for instance, to detect micrographic signs of plastic deformation before the 12-percent threshold found by the author.

V. Electrolytic Reduction

The problem of obtaining very thin specimens arises in the technique of radio crystallography by transmission.

The thinning by mechanical method does not permit a reduction beyond 0.5 mm and presents the danger of modifying the structure and texture within a minute fraction of that thickness. This danger is especially to be feared for pure aluminum which is very sensitive to cold working, and of course also for diluted solid solutions. It has even been encountered for the alloy Al - 7-percent Mg cold worked (34).

Electrolytic polishing offers a convenient means of achieving thinning without modifying the structure. In any case - in view of the fact that the dissolution of several tenths of a millimeter requires a long duration of electrolysis - one must seek protection from the thermal effect when the latter is liable to modify the structure, as is always the case in alloys with precipitation by tempering. In most cases, the specimens are - owing to their small dimensions - subjected only to a very insignificant current (at least in case of the acetic-perchloric bath which is the most recommendable one where considerable quantities of metal are to be dissolved) so that a dangerous heating up need not be feared.

In applying the method of polishing without modification, one must remember, as a general rule, that the dissolution is produced with greater speed on the edges of the anode on one hand, and on the region at the level of the electrolyte on the other. Consequently, one should give to the specimen dimensions slightly larger than those required for the intended final utilization. The more marked dissolution at the boundary surface between bath and atmosphere is easily avoided by making it take effect not on the piece to be treated but on the conductor (aluminum wire)

which connects it with the electric circuit. It suffices therefore if that conductor is so large that no reduction could occur. If that is not possible, one will take the precaution to displace the assembly, from time to time in the vertical plane.

The more rapid attack on the edges of the anode involves, as a consequence, a more noticeable dissolution on the periphery than toward the center. The difference is particularly distinct at the lower end of the test specimen where it is always slenderer than elsewhere (this is the effect which is put to use in the preparation of extremely fine points (29), another application of electrolytic polishing).

For making the dissolution uniform, one can contrive special arrangements such as a frame or support for the specimen which protect the edges or conduct the current to all points of the surface exposed to the bath (18).

For the taking of X-ray pictures by transmission through aluminum and its alloys, the electrolytic thinning does not present any difficulty since a thickness of one or two tenths of a millimeter is amply sufficient. This does not hold true for the heavy metals, but their case is not to be considered in this publication.

Recently, R. D. Heidenreich perfected at the Bell Telephone Laboratories the technique of dissolution of aluminum and aluminum copper by electrolytic polishing in order to prepare specimens which could be traversed by the electron beam of the electron microscope. Not only does this preparation eliminate the replica of aluminum but the images obtained are different from customary electronic micrographs in that the phenomenon of diffraction of the electrons plays a role (19).

Heidenreich has applied his technique for studying the structure of cold-worked aluminum and of a heat-treated 4 percent copper alloy. One can find the detailed description of the tests and their results in the original memorandum. Here we shall limit ourselves to giving some information on ultrathinning which will, besides, have to be perfected, as the author himself admits. Doubtlessly the method may be applied also to all metals and alloys suitable for being polished electrolytically.

The metal is rolled in sheets of about 0.13-mm thickness and undergoes the desired treatments of recrystallization, annealing, dissolving, etc. A fragment is polished electrolytically to eliminate the superficial inclusions and oxide; then one cuts off a disk of about 3-mm diameter. This disk is placed in the apparatus schematized in figure 1A which consists of a cylinder made of "teflon" (polytetrafluorethylene manufactured by du Pont de Nemours and characterized by its high resistance to acids and to organic solvents). The specimen is kept pressed against the circular surface of the cylinder by means of a cap, likewise of teflon,

which covers part of the disk in such a manner that only its central region is exposed to the electrolyte. The electric contact is obtained by means of a platinum wire traversing the stand to where it touches the specimen.

For aluminum, the electrolyte employed is derived from the formula indicated by Waisman and contains: methyl alcohol (1 part) - concentrated nitric acid (1 part), and, in addition, a trace of hydrochloric acid (1 cm³ of concentrated HCl for 50 cm³ of the preceding mixture). In comparison to Waisman's electrolyte, this latter electrolyte polishes under a weaker voltage and causes less discharge of gas. Moreover, the hydrochloric acid diminishes the tendency to formation of oxide.

The polishing is performed in a bath of 100 cm³, with a cathode of pure aluminum. The electric conditions are: a voltage of 5 v at the cell boundaries and an intensity of 1 amp. The position of the anodic support is the one indicated in figure 1A (long cylinder axis horizontal). The surface of the disk is at a distance of about 12 mm from the cathode and near one of its edges in such a manner that it can be illuminated by a light source and observed at an enlargement to four times its diameter.

The first stage consists in going on with the electrolysis until the thickness in the central region is reduced to about one half. The support is then rapidly withdrawn and plunged into running water, then rinsed in methyl alcohol. One takes off the cap, places the disk on a wire gauze, and shakes it in a mixture of equal parts of methyl alcohol and acetone. Then it is dried on filter paper. The polished surface must be very brilliant and clean. At this stage, the disk has the profile shown in 2, figure 1B. This profile is produced by the diminution of density of the current from the center to the edge at contact of the isolating cap.

The specimen is put back into the support, the unpolished surface turned toward the bath. During the new electrolysis, the surface is carefully observed under the magnifying glass and as soon as the first penetration has been ascertained, the support is pulled back and plunged into water. The sooner the electrolysis is stopped after formation of the first hole, the slenderer will be the utilizable part of the disk. One then repeats the rinsing operations described above. The profile of the specimen corresponds to 4, figure 1B; 5, figure 1B, shows the front view of the disk.

Examination at 50-x magnification shows the specimen to be perforated by several holes. Its manipulation is relatively easy owing to the rather thick edges.

The region surrounding each hole is sufficiently thick to permit examination by transmission in the electron microscope. Thus a very small part of the total surface can be utilized.

Obtaining convenient specimens is not always easy; one must frequently prepare five disks in order to obtain one satisfactory one. The main difficulties indicated by Heidenreich are the following:

(a) Insufficient thinned region is obtained. This is the case if the polishing has not been stopped soon enough.

(b) Roughnesses of the entire surface causing variations in thickness. The reason lies in unfavorable polishing conditions.

(c) Presence of a film of hydrous oxide. It is the result of a reaction of the acid on the metal during the rinsing. One can reduce it by pulling the disk back out of its support as quickly as possible. At the examination in the electron microscope one sees that the oxide layer penetrates into the holes; it is generally very transparent and frequently of granular appearance.

(d) Deformations of the thin regions due to gaseous discharge. One will have to reduce this discharge to a minimum, especially in the case where the specimen is used for study of plastic deformations.

The main disadvantage of this method of thinning is the smallness of the utilizable surface. In order to prepare larger surfaces, one would have to have an exact criterion of the instant at which the electrolysis must be stopped. Actually it is quite obvious that, as soon as a hole has been perforated, the very thin regions which surround it are rapidly dissolved.

Nevertheless, this technique has made it possible for Heidenreich to do very important work, such as the investigation of polygonization.

We want to mention one point which the author does not seem to have considered: that of the heating up of the disk. With its prescribed initial thinness, the specimen must heat up considerably during the electrolysis which may possibly lead to modifications of its structure, particularly detrimental if the investigation is concerned with electronic diffraction and micrography of plastically deformed aluminum. We have seen before that the temperature factor is almost always neglected by the authors who use electrolytic polishing and employ precisely those techniques where heating up will occur with certainty (bath operating at high intensity) and must well exceed the heating observed in the acetic-perchloric bath for which we found a temperature of 50° F at least on the surface of the anode.

SUPPLEMENT 1⁷

Very recently, R. Castaing and P. Laborie (C.R. 237, 1953, p. 1,330) utilized, with several modifications of details, the method of electrolytic thinning of Heidenreich, and have greatly improved the results by carrying out first a partial reduction, then an ion bombardment.

The authors have attempted to obtain at the same time an extensive usable surface and a sufficiently small thickness to permit observation by electron microscope with a high degree of resolution.

Up to now the tests have been limited to the study of an alloy of Al and 4-percent Cu at various states of tempering. The recrystallized alloy, taken in the shape of a foil of 0.2 mm thickness is cut up into pellets of 3 mm diameter. Each of these pellets is polished simultaneously on both sides in the acetic-acid - perchloric-acid bath; the fluid is made to circulate so as to avoid the formation of gas bubbles. The edges of the pellets are protected by a ring of aluminum which limits the polishing to the central part. The specimen is strongly illuminated from one side and observed from the other side by means of a binocular microscope of high magnification. The polishing is stopped when a portion of the surface begins to become translucent which corresponds to a thickness of 0.05 to 0.1 micron. In view of the fact that the velocity of anodic dissolution is of the order of 0.05 micron per second, it is difficult - but not impossible - to avoid formation of a hole in the thinned part, just as with the original method of Heidenreich.

Castaing and Laborie thus obtained on a surface of several times ten square microns a thickness sufficiently small to permit observation by electron microscope at the usable enlargement of 10,000. The figure 43 is an image thus obtained with the light alloy tempered 60 hours at 200° C.

⁷This supplement to part II was provided the NACA in a communication from O.N.E.R.A. dated May 17, 1954.

This method presents a certain number of defects:

(a) The electrolytic polishing may entail the formation of a superficial film of oxide and especially of a redeposit of copper - both sources of artifacts on the micrographs.

(b) The precipitates, for instance, of the phase $\text{Al}_2\text{Cu O}'$, less attacked than the matrix during the polishing, remain in relief, and the image will not correspond to the one which would be given by an ideal ultra-thin section.

(c) As soon as a hole has been formed, its borders are very rapidly dissolved, and only few specimens present an extensive usable surface.

It was possible to eliminate all these disadvantages with the technique of reduction by ion bombardment. The ion bombardment has already been recommended as a means of micrographic attack (see, for instance, D. M. McCutcheon and W. Pahl, Metal Progress, 56, 1949, p. 674). It has also been employed by C. Fert (C.R. 238, 1954, p. 333) for discharging the insulating particles and removing successive layers from the surface while the electron-diffraction diagrams are being taken.

The metallic specimen is, first, electrolytically reduced up to the stage which precedes the formation of holes. Then its two faces are subjected to the impact of a parallel and monokinetic cluster of ions of the energy 3,000 v. One examines the specimen periodically by electron microscope until a suitable reduction has been obtained.

Figure 44 which gives an example of the appearance obtained with a specimen identical with the one of figure 43 shows the excellent resolution at the convenient enlargement of 33,000.

With regard to electrolytic polishing, only the method of ion bombardment presents interesting advantages:

(a) The surface is extremely clean, without traces of oxide or foreign substances.

(b) The precipitates of the phase $\text{Al}_2\text{Cu O}'$ are practically on the same level as the matrix.

(c) The borders of a possible hole are not attacked more strongly than the rest of the surface which is very important.

(d) The speed of removal of matter is of the order of 0.5 micron per minute, and it can be regulated at will; hence the risk of heating up of the surface is eliminated.

(e) The procedure may be carried out inside the electron microscope itself which simplifies the manipulations and facilitates the examination.

There is no doubt that the employment of electrolytic polishing and ion bombardment combined is bound to permit the easy direct observation, at very high magnifications, of the structural evolution of aluminum alloys and probably also of other materials such as the refractory alloys for turbojets which are of interest for aeronautical construction.

On the other hand, the fact that the ion bombardment produces a true effect of polishing of the surface is interesting considered from the point of view of the mechanism of electrolytic polishing which is still an object of discussion. Thus, according to E. Darmon and I. Epelboin (C.R. 237, 1953, p. 501) the origin of the stage in the typical current/voltage curve of the phenomenon of electrolytic polishing (P. A. Jacquet, Trans. Electrochem. Soc., 69, 1936, p. 629) is to be approached to that of the stage of the discharge current in gases of low pressure. The polishing by ion bombardment is actually achieved without the formation of a film of oxide. This signifies that the analogous functioning of the electrochemical and of the ion method may be caused by the production of a tension on the surface of the metal which is sufficient for cold-working extraction of the electrons and ions of the grating.

THIRD PART

EXPERIMENTAL INVESTIGATION REGARDING USE OF AN AUTOMATIC

APPARATUS FOR ELECTROLYTIC POLISHING

The metallurgical laboratories use electrolytic polishing for two different purposes: on one hand, for the preparation of surfaces destined for particular research (plastic deformation, corrosion, surface properties, etc.); on the other hand, for examination of the micrographic structure. In the first case, the surfaces to be polished are generally extensive; they are, for instance, standard test specimens or single crystals which require polishing at all their points. Relatively large electrolytic cells provided with stirring devices, a cooling system, electric contacts, etc., are then indispensable. We have seen also that the character of the electrolyte must be taken into consideration.

If one deals, on the contrary, with a micrographic examination of the structure, the surface of the specimen is more or less limited. The form of the latter can, on the other hand, be standardized as is the case for the current controls being manufactured. One can therefore examine an apparatus and an electrolyte well adjusted for the polishing of these small surfaces. Nevertheless, two difficulties arise which complicate

the technique and cause certain laboratories to hesitate to use it. On one hand, the polyphase alloys rich in insoluble constituents require precautions if one wants to avoid relief effects which are injurious to the quality of the images and even to their interpretation. On the other hand, in order to achieve control of the structure, it is a fact that one is obliged to work with a relatively large area which makes it impossible to avoid relief effects. Under these conditions one must either polish the entire surface of the object or isolate electrically all portions not concerned. Both these means are difficult to apply correctly, and the quality of the polishing may leave something to be desired since the density of the current and the heating up (primary factors) are not easily controllable.

Added to these main difficulties is the necessity to take care of all the accessories required for setting up the apparatus. Finally, it must be said that with regard to the results obtained, whatever type of bath was selected, a "personal coefficient" of the operator comes into play, something which is necessarily admissible in a research laboratory but hardly compatible with accepted practices in large-scale production.

In order to remedy all these disadvantages which tend to limit the range of application of electrolysis and thus to deprive metallurgists of a valuable tool, designers of laboratory apparatus have conceived and put on the market devices for electrolysis which may complement machines polishing mechanically or be substituted for them entirely. At the moment there exist five types of such apparatus, two of which are constructed by the Danish firm H. Struers Chemiska Laboratorium.⁸

In our personal opinion one of these two types, the "Disa-Electropol," is especially well adapted to the current work of a laboratory of metallography.

In its present form, the Disa-Electropol (20 and 33) is suitable for micrographic polishing of common metallic materials, with the exception, however, of copper and its alloys. The manufacturer has just remedied this last limitation and will be able to deliver in the near future a model having certain modifications (more powerful pump, materials which better resist corrosion, inclined position of the electrolytic cell) which permit polishing copper alloys with the phosphoric electrolyte.

We have systematically explored the possibilities offered by the Disa-Electropol for the preparation of micrographic specimens of a large number of metals and alloys. Many among them are of interest in aeronautical technique (ordinary and special steels, refractory alloys, light alloys, titanium, etc); however, we shall limit ourselves here to aluminum and to the light alloys.

⁸Represented in France by the Société d'Applications de Procédés Electrolytiques.

I. Description and Operation of the Disa-Electropol

Figures 2 and 3 give an over-all view and the mounting scheme of the Disa which is fed by the 110 or 220 v alternating current.

On the block-shaped support 2 one places one of the three small-plate models (of plastic material) represented in figure 4, selected according to the shape and the surface one wishes to polish.

The specimen 3 is placed over the opening and pressed onto it by means of the spring contact 4. With the switch 9 placed in the position "Polishing" the circulation velocity of the electrolyte contained in the beaker 14 is regulated by means of the button 16 which operates the pump 15. This velocity must be sufficiently high to make the liquid flow out into the basin 1, passing through the second opening of small diameter of the small plate on which the specimen is mounted.

After the switch 7 has been put in the position v (voltage), the index of the timer 5 is placed in front of the graduation corresponding to the desired duration of electrolysis. This timer is started by pressing its pushbutton which simultaneously closes the electric circuit. The exact voltage is immediately adjusted to the desired amount⁹ by manipulating the control knob 10 of the potentiometer, and is read off the voltmeter graduated from 0 to 150 v. (The graduation 0 to 15 v corresponds to the circuit "etching," see farther below.)

Stopping of the timer cuts off the current. The specimen is immediately pulled out and its lower surface is washed under vigorously running water, rinsed with alcohol, and then dried in a jet of lukewarm air.

The duration of the electrolysis depends on the initial state of the surface. All the specimens described farther on have been prepared mechanically by abrasion up to 00 emery paraffined paper. An electrolysis of 20 seconds is sufficient to eliminate all scratches, and this duration has been adopted except for the smelting alloy Alpax with 12-percent silicon, the electrolysis of which must be limited to 5-7 seconds in order to reduce the relief of free silicon particles to a minimum.

The voltage at the cell boundaries is the only factor controlling the polishing. Its value, though, is almost completely independent of the character of the light alloy and of the polished surface. In general, this voltage is between 35 and 45 v with the electrolyte we have used, which furnishes the best results.

⁹It is recommended to determine first, by a preliminary test, the exact voltage desired.

II. Electrolytes

The directions for use of the "Disa-Electropol" provide for two types of electrolytes. The first which is suitable for ordinary and special steels, zinc, lead, silver, refractory alloys of Nimonic type contains 200 cm³ perchloric acid of a density of 1.20, 700 cm³ ethyl alcohol, and 100 cm³ glycerin. For castings and aluminum and its alloys, the preferably recommended formula is 35 cm³ perchloric acid of the density 1.61, 840 cm³ methyl alcohol, and 125 cm³ glycerin.

A short time before we undertook our systematic tests, E. Knuth Winterfeldt, a Danish author who contributed to the perfection of the Disa, published a new formula for an electrolyte which is apparently remarkably suited for dissimilar light alloys. This formula also appears in the most recent paper of the same author (20).

The new composition corresponds to the standard formula for steels in which the glycerin is replaced by butyl-cellosolve (glycol monobutyl ether).¹⁰ We have used this formula exclusively and adapted it also to polishing of uranium and zirconium (21).

III. Conditions of Electrolysis

As we said before, the voltage at the boundaries is the only variable requiring adjustment. It is also very high as identical results are obtained between 35 and 45 v. Nevertheless, it is preferable to work with the minimum voltage in order to limit the heating up. The latter cannot be completely avoided and is the more pronounced, the smaller the volume of the specimen. The duration of the electrolysis is too short to permit the temperature rise to cause an appreciable transformation of the structure visible under the ordinary microscope. However, it is not absolutely certain that the effect does not occur at the magnification for the electron microscope. In any case, this particular point would require verification which has not yet been made.

In the case of thin small plates, a good precaution for limiting heating up consists in inserting between the reverse surface of the specimen and the spring contact an aluminum block which will dissipate part of the heat given off.

As a consequence of the smallness of the surface to be polished - at the most 1 cm² - the density of the current is very high (150-175 a/dm²) which explains both the very short duration of operation and the

¹⁰This solvent, very much in use in the United States but less common in France, is on sale at the Société Minerals et Alliages, chemical-products department.

considerable heating up. This density of current is at least 20 times as high as in the case of the conventional acetic-perchloric bath where, as experience has shown, the surface of the anode may attain during the electrolysis a temperature of the order of 60° . However, in this last case the solution is not agitated; the rapid circulation is a factor which - on the Disa - limits to a certain extent the heating in the course of polishing.

The velocity of anodic dissolution has not been determined experimentally for aluminum; however, we have data for steels which show that in 20 seconds a thickness of 10 microns is removed which corresponds to an electrolysis of 2 minutes 30 seconds duration in the $1,000 \text{ cm}^3$ acetic-acid - 50 cm^3 perchloric-acid electrolyte, with the normal density of current of 15 to 20 amp per dm^2 .

IV. Electrolytic Attack

Like all procedures of electrolytic polishing, the treatment with the Disa reveals the dissimilar structures on the surfaces without supplementary attack. Nevertheless, employment of the usual reagents is necessary in order to differentiate certain phases, make the grain boundaries appear, or make the traces of the constituents better visible. Circumstances are known where certain chemical reagents produce parasitic effects of etching whose aspects confused surface effect (pitting, intergranular corrosion). For this reason, detection of the fine dispersions in the mass or on the boundaries of the grains is much more certain if one makes an examination under high magnification of the surface which has been subjected only to polishing.

The Disa offers the possibility of subjecting the polished specimen to an electrolytic attack, using three different methods of operating which seem very interesting but have not yet been systematically explored for the micrography of light alloys.

The two first methods of attack consist of utilizing the polishing bath and a special circuit functioning when the contact 9 (fig. 2) is put on the position "etching." In this circuit, the graduations of the voltmeter and the ammeter do no longer correspond to 0-150 v and 0-1.5 amp, but to 0-15 v and 0-0.15 amp.

First method: Three or four seconds before the end of polishing one switches abruptly from "polishing" to "etching." The voltage at the boundaries which had been 40 to 45 v changes to 3 to 4 v.

Second method: The electrolysis is continued to its normal termination, that is, one awaits the stopping of the timer which cuts the current off, before changing to the position "etching." The index of the timer is placed on the position 3 or 4 and the connection is established.

These two methods are not equivalent because the first involves the changing of the switch to the position "off" which simultaneously cuts off the current and stops the agitation in such a manner that, when the circuit is again established, the electrolyte does not yet circulate at its normal velocity. On the other hand, the exact duration of the attack is not very well defined. For these reasons the second method frequently yields results which are more easily reproducible and more uniform on the surface.

The third technique of anodic attack puts an auxiliary apparatus to work, represented schematically in figure 5, which is adjusted by means of the control knob 11 (fig. 2). Here any electrolyte whatever may be employed, for instance, an anodic oxidation bath. One starts by placing the switch 9 and the selection switch 12 on the positions "etching" and "on," respectively. The two lamps 8 and 13 light up and the pump operating during the polishing is disconnected from the circuit.

The polished, washed, and dried specimen is placed on the plate (polished surface upward) which is connected to the positive pole. For light specimens, it will be of advantage to replace this plate by a strong clamp which ensures a better electric contact.

V. Results

All light alloys tested on the Disa have given excellent results from the viewpoint of micrographic examination. Table II gives the name, composition, and condition of the various specimens examined. It refers, moreover, to the corresponding figures. We shall successively review each of these alloys, specifying possible particulars which deserve to be pointed out.

Commercial aluminum.- This metal is typed as 99.7 percent (principal impurities iron and silicon). The specimen has been sectioned from a raw-cast ingot.

Figure 6 shows that the structure contains elements of constituents lined up on the intergranular boundaries and of small spherules distributed in the interior of the large grains. Under slight magnifications, these circular elements give the impression of being micropits, or cavities, but this impression disappears when the magnification is increased (figs. 7 and 9). One deals here actually with spherules of eutectic structure; it is difficult to make their presence evident after mechanical polishing; however, P. Rocquet (22) has already found them in certain commercial aluminum polished electrolytically in an acetic-perchloric bath. It is generally accepted that these spherules are the ternary eutectic $\text{Al-Al}_4\text{FeSi}_2\text{-Si}$.

It is gratifying to point out the absence of relief and of etching around the spherules and the intergranular constituents; etching around the grain boundaries is a rather frequent polishing defect in electrolytes concentrated in perchloric acid or consisting of mixtures of sulphuric and phosphoric acids.

Cast-aluminum - copper alloys.- We investigated the three alloys with 8, 10, and 12 percent copper, all cast in a chill mold. The eutectic $\text{Al}-\text{Al}_2\text{Cu}$ is always perfectly dissolved and does not show any relief, whatever the magnification may be (figs. 10 and 11).

Moreover, the copper film which is frequently deposited during the washing of the polished surfaces in the acetic-perchloric bath does not appear here.

Aluminum - copper (8 percent) - manganese (1 percent) alloys.- The addition of manganese causes a constituent $\text{Al}-\text{Cu}-\text{Mn}$ to appear in the structure of the small plates which likewise do not show any effect of etching or of relief (fig. 12). As an example, we have reproduced (fig. 13) the micrograph of the same specimen obtained in a bath of two parts acetic anhydride and one part perchloric acid, considered satisfactory at the time (1944). The comparison makes any commentary superfluous.

TABLE II.- SPECIMENS POLISHED ON THE DISA-ELECTROPOL

<u>Name</u>	<u>Composition</u>	<u>State</u>	<u>Figures</u>
Commercial aluminum	0.3 percent of impurities (iron and silicon)	Raw-cast ingot	6, 7, 8, 9
Aluminum-copper alloys (C.F.M.)	With 8 percent copper	Cast in chill mold	10
	With 10 percent copper	Cast in chill mold	
	With 12 percent copper	Cast in chill mold	
Aluminum-copper-manganese alloy (C.F.M.)	With 8 percent copper and 1 percent manganese	Cast in chill mold	12
Alloy Al-Cu-Mg-Si (C.F.M.)	Cu (4 percent) - Mg (0.2 - 0.4 percent) - Si (0.25 percent) - Ti (0.15 percent)	Cast in chill mold	14
Fortal (C.F.M.)	Cu (4 percent) - Mg (0.6 percent) - Si (0.5 percent)	Welded	15
Alloy Y (C.F.M.)	Cu (4 percent) - Ni (2 percent) - Mg (1.5 percent) - Si (0.5 percent)	Cast in chill mold	16, 17
AG7 (T.L.H.)	With 7 percent magnesium	Continuous case billet 1/2. Cylindric containers hot-drawn then cold-formed	18 to 29
Alpax (C.F.M.)	With 13 percent silicon	Refined ingot	30, 31, 32
Zicral (French aluminum)	Al-Zn-Mg (plus addition of Cr)	Rolled angle ions	33
T60 (T.L.H.)	Al-Zn-Mg (plus addition of Cr)	Rolled sheet aged 6 years after tempering	34
75 ST (Alcoa U.S.A.)	Zn (5.6 percent) - Mg (2.5 percent) - Cu (1.6 percent) - Mn (0.2 percent) - Cr (0.3 percent)	Heat treated sheet	35
R.R. 58 (Hispano-Suiza)	Al-Cu-Ni-Mg-Fe-Si-Ti	Blade forged and hot worked	37, 38, 39
Vival (C.F.M.)	Al-Mg (1 percent) - Mn (0.2 percent) - Si (0.7 percent)	Welded, annealed. Treated at 550°, aged at 175°	40, 41, 42

Aluminum-copper magnesium-silicon alloys.- One of them contains, in addition, 0.15 percent titanium. Electrolytic polishing reveals that the insoluble constituents form a eutectic in the state "cast in chill mold" (fig. 14) and are dispersed in small plates after welding (fig. 15). In order to distinguish the character of these constituents, it would be necessary to follow the polishing up with a suitable attack.

Alloy Y.- This alloy which contains chiefly copper, nickel, and magnesium, polishes excellently in spite of the fineness of the elements of the constituents (figs. 16 and 17). Here again a special attack with conventional reagents would be required for identification of the various constituents.

Aluminum - 7 percent magnesium alloys.- The micrographs here presented are extracted from a long investigation undertaken at the occasion of an appraisal by experts of the causes of the explosion of containers made of alloy AG7 containing air compressed under 200-kg pressure and exposed to marine atmosphere (30).

We intend to provide subsequently certain details regarding this investigation to the extent that it touches upon the fundamental problem of corrosion under tension. For the moment, we shall be content to give examples of structures observed on several specimens polished on the Disa-Electropol.

Figures 18 and 19 are two micrographs taken at the same place in a section of the billet used as basic material for the manufacture of the cylindrical containers. The first shows the elements of the primary constituent Al_3Mg_2 which are rather large and rather numerous in this 7-percent alloy. The second shows the interdendritic network rich in magnesium which connects the fragments of the primary constituents by a sort of bridge. On these two views, one will note the presence of black spots which are porosities or more probably inclusions of oxide, frequent in alloys relatively rich in magnesium.

The method of manufacturing the containers consists in hot spinning, then cold forming of the rough shapes thus obtained. No special precautions have to be taken regarding the cooling off which follows the hot working so that a small quantity of magnesium may leave the solid solution in the form of precipitation which is preferably localized on the grain boundaries. This aspect is visible on figure 21. One will note the extreme fineness of the elements of the precipitation and their irregular distribution - a very important phenomenon to which we shall return later.

Another interesting particular is the presence of rare intergranular microcracks, associated or nonassociated with primary constituents or with some of the precipitate. Figure 20 shows such a crack. This defect

is in accordance with recent results of British authors which show that cold deformation of an alloy with 7-percent magnesium, made sensitive to corrosion under tension by tempering, may produce intergranular cracking (23).

Since the reports of Lacombe and Beaujard, anodic oxidation has become a very effective means of revealing the grain boundaries of an Al-Mg alloy subjected to tempering. According to these authors, the anodic oxidation attacks selectively the grain boundaries, even if the optical microscope is not capable of revealing the slightest trace of precipitation. Moreover, the grains themselves become visible owing to the interference colors of the aluminum film since the optical characteristics of the latter vary according to the orientation of the crystals supporting it. Thus the various grains may be distinguished by examination in polarized light when their boundaries are not clearly defined (figs. 22 and 23).¹¹ Figure 24 shows, on the contrary, a continuous intergranular system.

An acid attack (hydrofluoric acid diluted with 5-percent phosphoric acid) reveals likewise the grain boundaries of the alloy that has been tempered or cooled off rather slowly from a high temperature (fig. 25). In contrast, the alloy tempered in water starting from 460° does not show an intergranular attack, either by anodic oxidation or by immersion in the diluted hydrofluoric acid. Besides, for this case, electrolytic polishing does not make the irregular precipitate appear either; an example has been given in figure 21.

The relationship between the exact character of the grain boundaries, on one hand, and their visibility by electrolytic polishing, anodic oxidation, or acid attack, on the other hand, seems to us very obscure. The numerous investigators who have dealt with this question - because it is of interest as an explanation of the phenomenon of corrosion under tension of the aluminum-magnesium alloys - are unanimous in declaring that if the micrographic structure of the specimen contains a continuous intergranular system, this latter corresponds to a precipitate, likewise continuous, of the constituent rich in magnesium. Their conclusion is based on micrographic examinations of mechanically polished specimens which have then been attacked by an acid solution (hydrofluoric or phosphoric). Actually, this method of detection implies a genuine corrosion of the boundaries since our own observations show that in the electrolytically polished state these boundaries are sometimes discontinuous and become continuous only after anodic oxidation or acid attack. The fact that the electrolytic

¹¹The anodic oxidation is carried out in a solution of phosphoric acid to which butylcellosolve has been added (106 cm³ concentrated PO₄H₃ - 40 cm³ water - 40 cm³ butylcellosolve) under a voltage of 40 v. The specimen is agitated by hand through the entire duration of electroanalysis (6 to 12 minutes).

polishing reveals at the grain boundaries very small, clearly separated segments would tend to prove that one deals here with a chemical heterogeneity shown by its rapidity of anodic dissolution which is different from that of the solid solution, for the same reason for which the polishing directly makes the primary constituents appear cast. However, the ordinary micrography neither confirms nor invalidates this interpretation since, in fact, each small intergranular element must be of a size below the resolution limit and, consequently, is not visible under its normal form and size at the magnification 2,000. Whether the electron microscope is capable of bringing us the desired proof - that we shall see in another investigation in the near future.

If the aluminum-magnesium alloys are heated at high temperature and, consequently, do not show any precipitation (or, more exactly, transformation) on the boundaries, it is very difficult to make these latter appear. So far, the method of anodic oxidation producing differential colorations of the aluminum film is the only possible one for evaluating the grain size.

However, it is now possible to show the grains by utilizing the circuit etching of the Disa, according to the two methods of operation previously indicated. The first which consists in passing from "polishing" to "etching" 2 or 3 seconds before the end of the normal duration of polishing produces a very slight etching of the grains without preferential attack on the boundaries, nor on the primary constituents either, even when these boundaries are attackable by hydrofluoric acid or by anodic oxidation. (See, for instance, fig. 28 which corresponds to the cross section of a specimen the grain boundaries of which are nonhomogeneous. The exterior surface of that specimen is shown in fig. 21.) Nevertheless, this first method of operation does not always give a perfectly uniform attack on the entire polished pattern.

The second method appears to be more interesting and would deserve a systematic presentation which has not yet been made. Here, the polishing is terminated when the circuit is cut. One then passes at once to "etching," and one waits a few seconds until the circulation of electrolysis has assumed its normal value. At this moment, one moves the index of the timer 2 or 3 seconds ahead, and one closes the circuit. With this method, the intergranular boundaries are always revealed, whatever the structural state of the alloy may be, but the aspect of the attack is different as shown by comparison of the figures 26 and 27. It is remarkable that the boundaries appear more heavily attacked precisely in the case in which the alloy is heat treated, where they cannot be detected by an acid attack; however, they do not appear perfectly continuous. On the contrary, with the alloy where the grain boundaries are dissimilar, the results obtained with electrolytic attack is reasonably identical with the results after an attack with hydrofluoric acid. (Compare, for instance, the figs. 25 and 26.)

To end with the alloy with 7-percent magnesium, we give the micrograph (fig. 29) which is of particular interest because it has been obtained with the same electrolyte as the one used with the Disa, but in a normal cell under a voltage of 60 v.¹²

The difficulty lies in preventing a very considerable heating up of the surface which may cause etchings or oxidations. Doubtlessly, an energetic agitation would be necessary. For the micrograph of figure 29, the electrolyte was stationary and the sample was agitated by hand.

Aluminum-silicon alloys.— Many metallographists believe that the aluminum alloys containing a rather large amount of silicon, for instance the cast alloy with 13 percent (alpax, silumin), cannot be polished electrolytically for micrographic examination; the silicon, being insoluble, remains in relief while the solid solution surrounding it is more strongly dissolved than elsewhere.

The results obtained with the polishing method of the S.N.C.A.S.O. (electrolyte of Matte and David) the details of which we have given in our book, already have shown that the eutectic Al-Si is perfectly dissolved on the polished surface. The Disa likewise furnishes very correct results, permitting in particular, the verification of the degree of purity in the presence of sodium. Figure 30 shows as an example a section where large fragments of silicon subsist in the alloy. These fragments are seen under higher magnification in figure 31 where one distinguishes moreover, in the midst of the solid solution, two or three elements of a foreign constituent, probably Al_2Cu . Finally the figure 32 corresponds to another portion in the same specimen, cut parallel to the first, but at a distance of 22 mm; the eutectic is relatively fine, and at slight magnification no massive particle of silicon is visible.

The Disa-Electropol seems therefore very useful for consistent current control of the degree of dispersion of the silicon in the solid solution, and for the detection of foreign elements.¹³

¹²The total intensity is higher than with the Disa, because of the larger anodic surface exposed.

¹³Quite recently, the introduction of a special commutator (designed to invert the direction of the current at a given moment during the electrolysis) into the electric circuit of the Disa permitted E. Knuth Winterfeldt to obtain remarkable micrographs of alloys rich in silicon (31).

Figure 45 (which is supplement 2 furnished NACA in May, 1954) gives an example of a micrograph thus obtained concerning a cast alloy containing 12.85-percent Si - 0.51-percent Fe - 0.38-percent Mn. Comparison of this figure with the figures 30, 31 and 32 shows the considerable improvement in the results.

Aluminum-zinc-magnesium-type alloys of high strength.- These alloys have been studied for a long time in the United States, in Great Britain, and in France. In our country, research has led to two commercial alloys, Zicral (of French aluminum) and T60 (from the Wire Mills and Rolling Mills du Havre). In America, Alcoa manufactures the alloy 75S (5.6-percent Zn, 2.5-percent Mg, 1.6-percent Cu, 0.2-percent Mn, and 0.3-percent Cr), and English aeronautical construction is interested in the alloy D.T.D. 693 which contains as the principal elements 6-percent Zn, 3-percent Mg, and 1-percent Cu.

The corrosion under tension of this type of alloys has posed problems which have retarded their practical utilization for a long time. These problems now appear to be solved.

In order to diminish the tendency toward corrosion under tension, certain elements are added to the Al-Zn-Mg alloy, among them chromium which is introduced from a concentrated parent alloy.

The micrographic examination of the Zicral produced about 1946, 1947, and of the alloy T60 (rolled sheet, heat treated at 470° on April 13, 1944 and aged under atmospheric conditions until the date of the examination, July 1950) reveals the presence of fragments of the constituent rich in chromium. These fragments are slightly less large but more abundant in T60 than in Zicral, as indicated in the two figures 33 and 34. The constituent rich in chromium appears in the form of small plates with very rectilinear contours; the largest are more or less fissured, without any relief with regard to the solid solution.

For comparison, figure 35 shows the surface structure of a sheet of the American alloy 75S which has been heat treated. The plates of constituents rich in chromium are present here also but they are very infrequent and of extremely reduced size.

Without any doubt, the abundance and dimensions of the constituent Al-Cr explain the bad performances (under fatigue, for instance) of the Zicral and the brittleness of the T60 (the sheet breaks at the first attempt of bending) possessing the structure shown in the micrographs supplementing this report.

Since the time when these specimens were prepared, manufacturing methods have been greatly improved, and from the micrographic viewpoint the French alloys are perfectly comparable to the American alloy 75S.

Alloy R.R. 58.- The alloys of the type RR (or hiduminium), produced by the English company High Duty Alloys, Ltd., are light alloys containing 0.5-to 5-percent copper, 0.2-to 1.5-percent nickel, 0.1-to 5-percent magnesium, 0.6-to 1.5-percent iron, 0.2-to 5-percent silicon, and a little titanium (up to 0.5 percent).

The RR 58 has been especially designed for the manufacture (by forging and hot extending of the blades of the air compressors for modern turboreactors. The micrographic structure of one of these blades has been investigated, and this case constitutes a very good example for the possibilities for the nondestructive examination of manufactured samples offered by the Disa-Electropol.

It is actually possible to locally polish the desired area, provided the latter can be placed on the stage of the microscope. The flat portion polished in two adjacent places 1 and 2 (fig. 36) was selected. The figures 37, 38, and 39 show the very good solution of the constituents under all magnifications. The presence of a system forming a sort of "veining" in certain large grains is particularly interesting since this aspect seems to indicate a state of polygonization rendered visible owing to the precipitation of a very fine constituent on the subboundaries. We intend to determine this structure exactly by making a micrograph by means of the electron microscope.¹⁴

Alloy "Vival".- This alloy manufactured by the Compagnie Francaise des Metaux contains 1-percent magnesium, 0.2-percent manganese, and 0.7-percent silicon.

Its structure, in welded state (fig. 40), shows constituents in the forms of small plates of a light color, and others less regular and darker, probably rich in silicon.

In the same alloy treated 20 minutes at 550° and then 2 hours at 175°, the light-colored constituents are less numerous and of smaller size. Observation under high magnifications (fig. 41) indicates clearly that they are in the state of solution. The grain boundaries are invisible in case of polished condition and appear only after hydrofluoric attack (fig. 42).

VI. Conclusions

The examples given here, selected from the most typical light alloys, show the quality of the micrographs obtained with the automatic apparatus Disa-Electropol. With this new method, the main objection raised so far against electrolytic polishing of heterogeneous aluminum alloys, namely, the exaggerated attack of certain constituents, is no longer tenable. One must concede that the acetic-perchloric electrolyte sometimes yields equivalent results, but this requires special precautions hardly compatible with the requirements of a control laboratory.

¹⁴Our specimens, examined by X-rays by A. Guinier, did not show the typical indications of polygonization. Doubtlessly the method is here insufficient because of the smallness of the polygonized domains.

The possibilities offered by the Disa concerning the electrolytic attack also constitute a valuable asset; systematic investigation of this advantage still has to be carried out, chiefly regarding demonstration of the grains; with light alloys, this leads sometimes to problems which are hard to solve.

VII. Appendix - Case of Electromicrographies

So far, our study has been limited to ordinary micrography, that is, to maximum magnifications of 2,000 to 2,500 diameters. The domain of electromicrography has not yet been entered.

The excellent results obtained with the optical microscope cannot be extended a priori to the domain of electromicrography. The greatest advantage of the electron microscope lies in the possibility of observation of ultra-fine structures, corresponding for instance to the beginnings of precipitation during tempering of supersaturated solid solutions. To get these structures clearly revealed, the base of the solid solution must be very well polished on a submicroscopic scale. As experience has shown, the electrolytes rich in perchloric acid generally permit obtaining a polish of this quality but we do not know whether the medium used with the Disa and the conditions of electrolysis which are peculiar to the apparatus will allow us to arrive at the same result. The test is worth a trial, but personally we would feel inclined toward answering in the negative. This opinion is based on certain observations published in the technical literature or set forth in private discussions.

We allude especially to the structures described by Geach and again found, besides, in high-purity aluminum by other specialists of electromicrography (24). The appearance (striations, cells, etc.) of the aluminum replicas appear to indicate a micro-attack on the metal, but one does not know yet whether this attack is characteristic of the electrolytically polished surface or whether it develops in the course of formation of the replica itself. In any case, it seems definitely demonstrated that appearances are characteristic of the metal base since they change from one side of a grain boundary to the other.

According to Geach (25), for the same type of aluminum electrolytically polished under the same conditions, the replica does or does not present the substructure according to the conditions of the anodic oxidation. Thus the cause would not lie in the method of polishing. Unfortunately, other experimentors sometimes find a substructure of the film, even with the conditions of polishing on one hand, and of the oxidation on the other apparently remaining identical in all cases.

In our opinion, this problem of the substructure revealed by the electron examination of the replicas has not yet been solved. The reasons for that are the following:

(1) The different appearances observed have not necessarily the same cause.

(2) For a certain technique of electrolytic polishing, minute modification of the conditions (the surface temperature, for instance, the duration of polishing, the age of the bath, etc.) may lead to an excellent polish or to a micro-attack not apparent under the ordinary magnifications, but detected by the replicas.

(3) An anodic oxidation is possible, either in the polishing bath or in the course of the final washing. The structure of this sublayer, more or less thin, on which the actual replica is going to form, introduces therefore an extraneous factor which is, by the way, difficult to control.

(4) It is known that the method of electrolytic polishing has a great influence on the chemical character of the obtained surface. As we have said before, the electrolytes with a base of phosphoric acid or of nitric acid lead with certainty to a polished but oxidized surface. The thickness, and perhaps also the structure, of the aluminum layer can be controlled only with difficulty in case of rich electrolytes. It is the presence of this layer which doubtlessly explains why certain authors have not been able to use the technique of Lacombe's etch figures on ultra-high-purity aluminum electrolytically polished in a phosphoric medium.

(5) At the present state of techniques, the electrolyte consisting of one part of concentrated perchloric acid and two parts of acetic anhydride seems best adapted to producing nonoxidized aluminum surfaces. Nevertheless, the possibility of oxidation is not excluded even in this case (effect of the temperature, of the age of the bath, of the density of the current, etc.). Certain authors have pointed out this possibility in the course of their research regarding the potential of dissolution or the formation of etch figures, but, to our knowledge, not one of them has indicated the exact conditions which permit avoiding it.

(6) The conditions for production of the replicas by anodic oxidation may be very well defined but only to the extent to which the physical chemical characteristics of the surface of the metal themselves are defined and reproducible. For instance, the amount of the initial current will not be the same for a perfectly bare surface as for a surface already partially oxidized.

(7) Electrolytic polishing of the surface of the specimen is almost always carried out after a mechanical preparation involving abrasion. Demonstration of the effects of this abrasion on the structure is difficult, perhaps because they are minute and concern only a slender layer

below the surface, or perhaps rather because we have not yet an appropriate reagent at our disposal such as exists for other metals and alloys. It is possible that the anodic oxidation constitutes precisely such a reagent in the structural roughness at the magnification of the electron microscope.

This enumeration of the possible causes for the substructure from replicas suggests systematic investigations the results of which will permit an improvement in the quality of the images and for that very reason an extension of the possibilities for electron micrography for the understanding of the structure of aluminum and of light alloys. The case of the specimens prepared with the Disa-Electropol deserves particular attention because the conditions for functioning of this apparatus are more readily controllable.

GENERAL CONCLUSIONS

The employment of electrolytic polishing in the preparation of test specimens intended for special investigations and for micrography has greatly developed during the last 3 years for aluminum and its alloys. It has indeed become an exception for research in this domain not to resort at some stage to this preparation, the manifold advantages of which are too well known to have to be recalled.

Nevertheless, the metallographist should not lose sight of the secondary effects of electrolytic polishing, quite especially heating up and anodic oxidation. From this point of view, the various techniques of electrolysis utilized in laboratories are not equivalent. Thus it cannot be a matter of unconcern whether to employ one or the other, assuming the polishing effect as one can appreciate it with the naked eye or under the microscope, to be the same.

Heating up interferes when the structure or texture of deformation of high-purity aluminum is to be investigated. Heating may also modify the electron micrography of alloys with structural hardening although in general this effect will be little marked, owing to the short duration of the electrolysis.

It was believed for a long time that no single method existed which would permit to electropolish aluminum of whatever grade purity, and all its alloys.

The systematic tests we have undertaken with the commercial apparatus Disa-Electropol seem to indicate that such a method does exist now. Actually, it is now applicable only to ordinary micrographic investigations.

Extension to electron micrography will have to form the object of special research. Already we know that the Disa-Electropol does not permit the preparation of surfaces larger than 1 cm^2 ; thus the other procedures remain of interest. The most general method, that is, the one which is suitable for any shapes and surfaces, for materials of various composition, is doubtlessly the one which employs the perchloric-acid - acetic-anhydride electrolyte. This method has, moreover, the advantage of polishing without causing an oxide layer thick enough to be harmful. Also, the low value of the current limits the heating up of the surface.

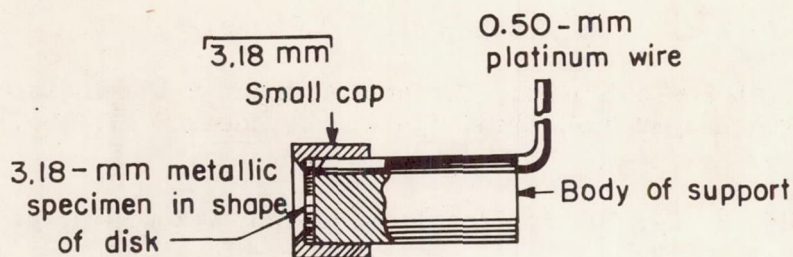
On the whole, the progress of electrolytic polishing in the domain of light metals and alloys has been substantial, as much as regards techniques as also applications at the laboratory. Nevertheless, it is desirable that both be further perfected. We want to point out quite particularly the achievement of ultrathinning, already begun in the United States, and the study of the problem of intergranular corrosion for which the combined use of electrolytic polishing and various methods of investigation - including the electronic probe of Castaing (32) - will perhaps permit us to understand at last the nature of the processes employed.

Translated by Mary L. Mahler
National Advisory Committee
for Aeronautics

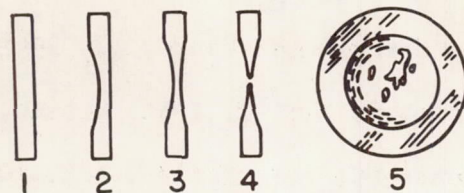
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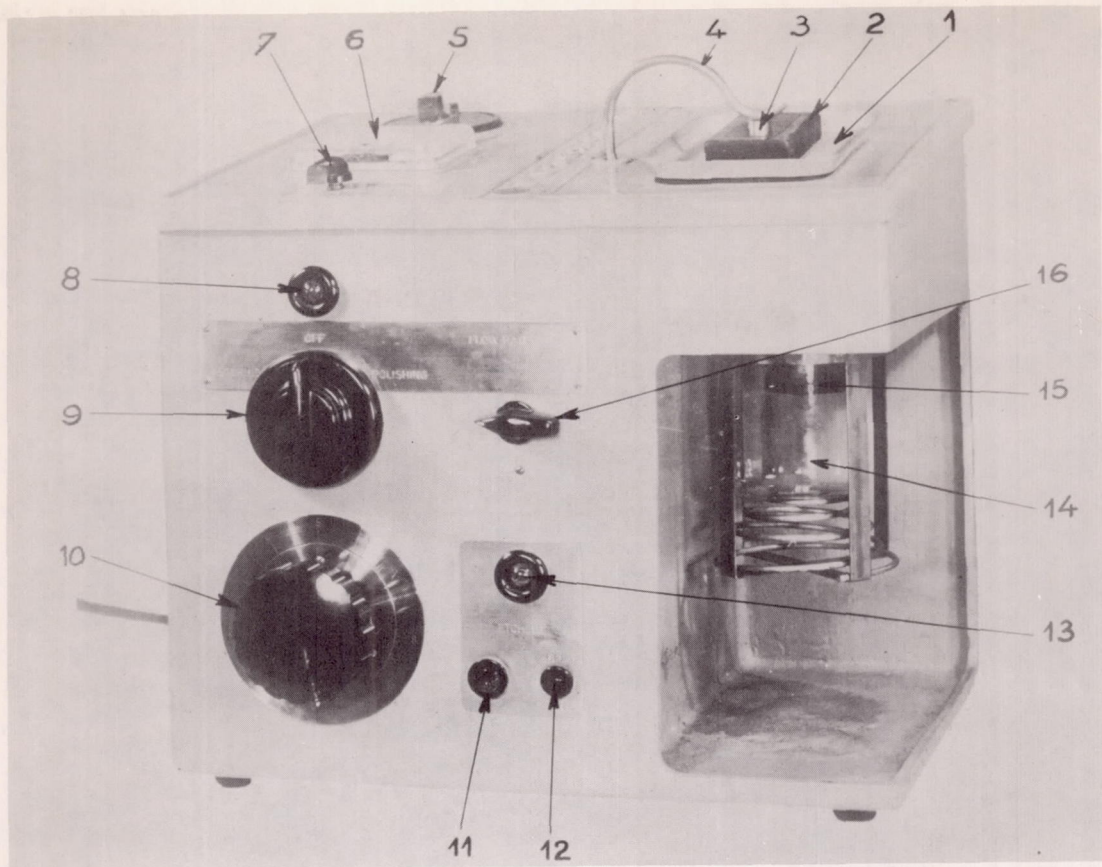
A. Support of specimen (in "Teflon")



1. Initial profile of disk (thickness 0.13 mm)
2. First surface electrolytically polished on the support
3. Disk is turned and the second surface is polished
4. Stage at which the thinning is stopped
5. Front view of the specimen at stage 4 showing the pits

B. Stages of thinning. (Taken from R. E. Heidenreich, Electron microscope and diffraction study of metal crystal texture by means of thin section, Journal Applied Physics, 20, 1949, p. 993.)

Figure 1.- Thinning-down of the specimens for electromicrography and diffraction.



1. Basin receiving overflow of electrolyte
2. Block support of specimen
3. Specimen placed on the block support
4. Electric spring contact
5. Timer 0 to 60 seconds, automatically cutting the current
6. Measuring apparatus graduated in volts and in amperes
7. Voltage-amperage switch
8. Control lamp indicating closing of the circuit
9. Interrupter and polishing switch
10. Potentiometer control button
11. Receptacle for plug for auxiliary etching device (see fig. 5)
12. Interrupter for auxiliary etching
13. Control lamp indicating connection of the device for auxiliary etching to the circuit
14. Beaker containing the electrolyte
15. Circulation pump for the electrolyte
16. Adjusting button for the pump regulating the circulation speed of the electrolyte

Figure 2- Disa-Electropol.

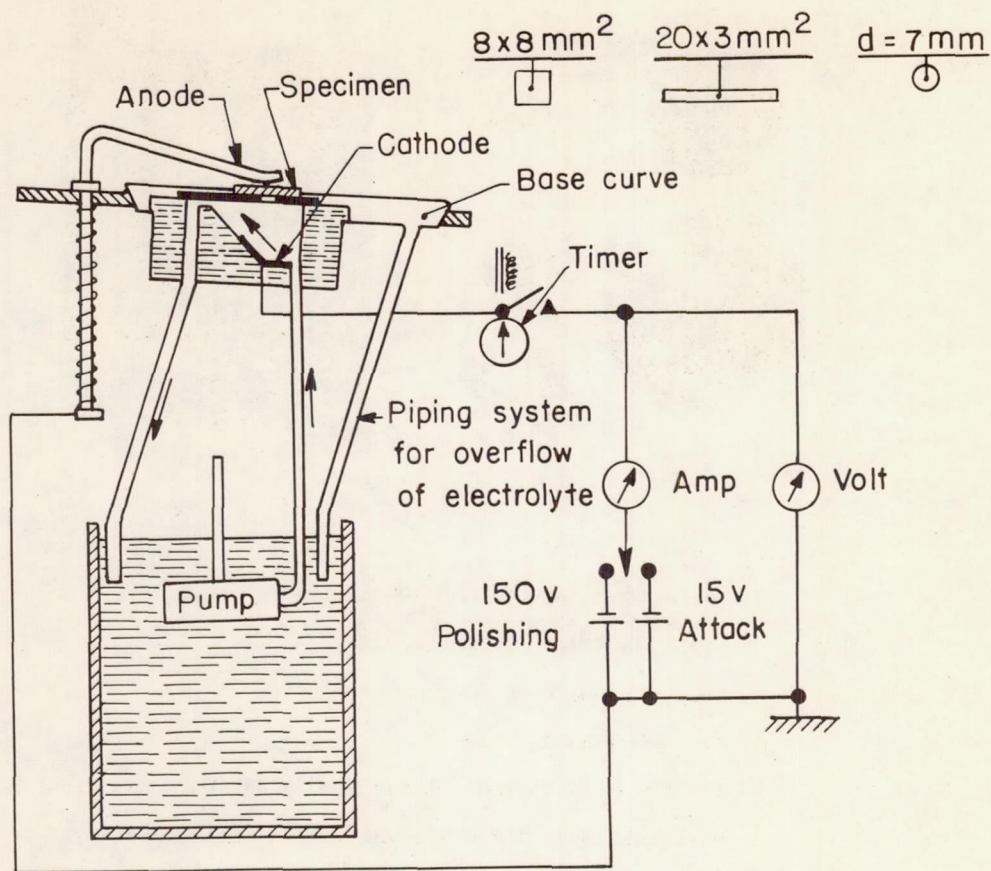


Figure 3.- Construction scheme of the Disa-Electropol.

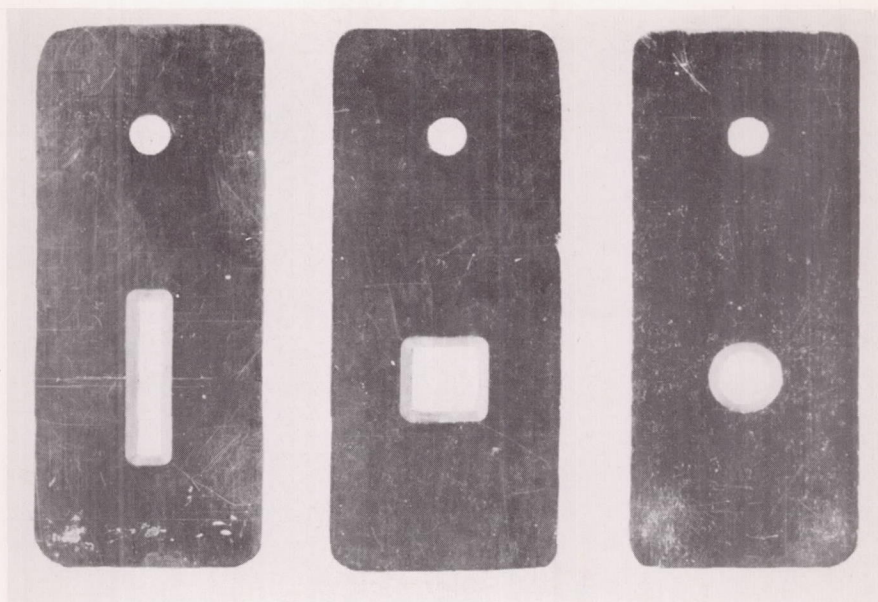


Figure 4.- The three models of sample support placed on the block 2 (fig. 2).

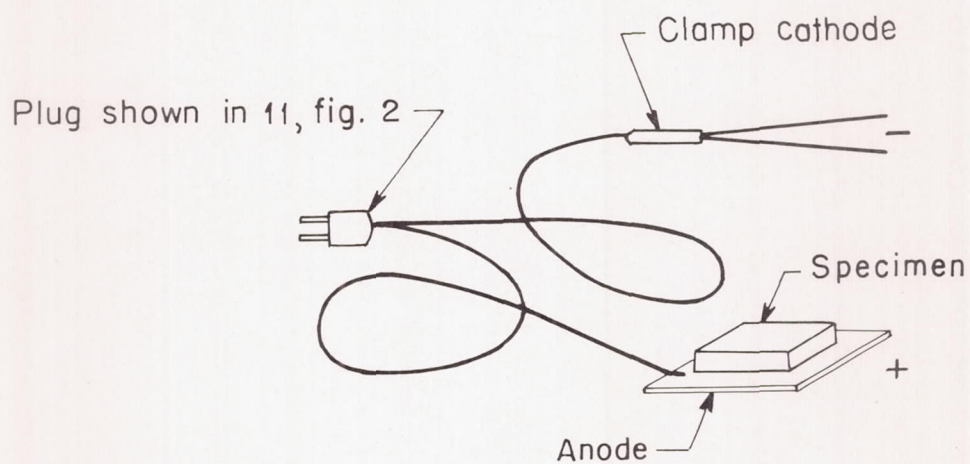


Figure 5.- Auxiliary device for anodic attack on the polished specimens.

99.7 percent aluminum containing Fe and Si; rough-cast.

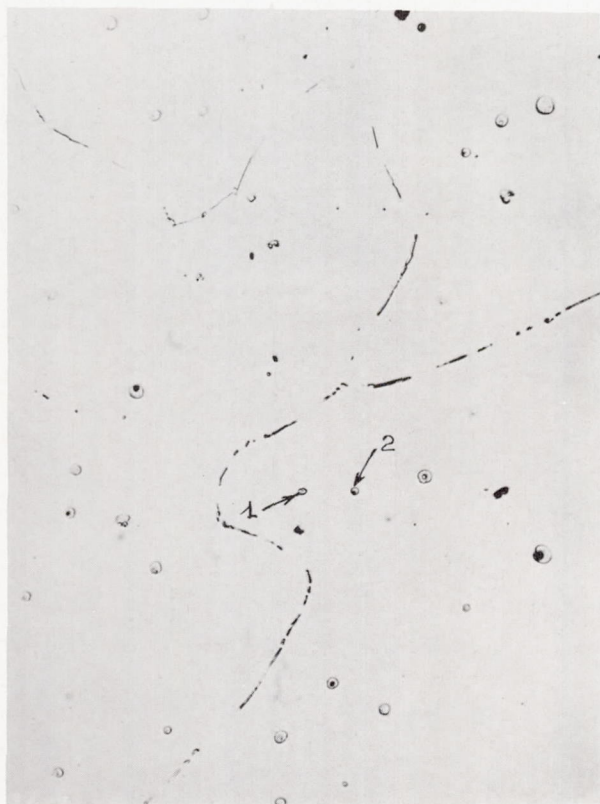


Figure 6.- Insoluble impurities on the boundaries and spherules of ternary eutectic $\text{Al}-\text{Al}_4\text{FeSi}_2-\text{Si}$. X160.

99.7 percent aluminum containing Fe and Si; rough-cast.

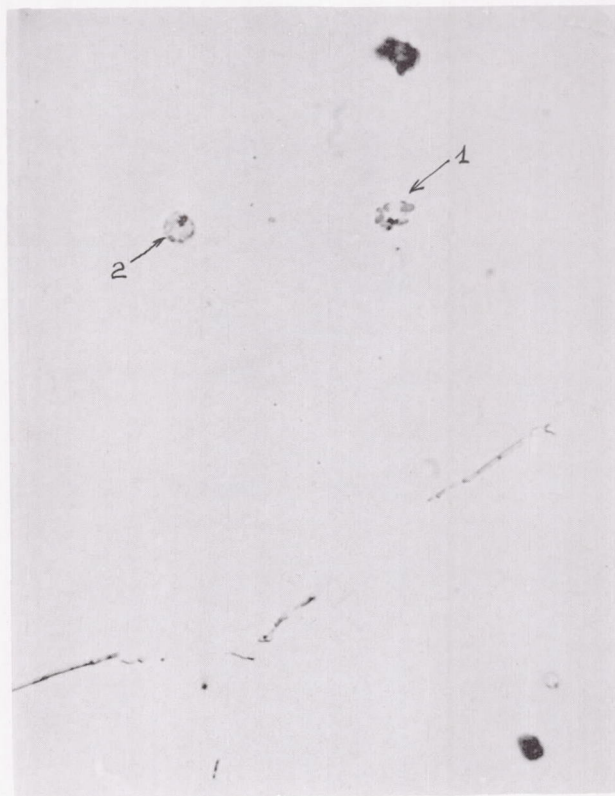


Figure 7.- Spherules 1 and 2 of figure 6. X630.

99.7 percent aluminum containing Fe and Si; rough-cast.



Figure 8.- Constituents in the grain boundaries. X2000.

99.7 percent aluminum containing Fe and Si; rough-cast.



Figure 9.- Spherule 2 of figure 7. X2000.



Figure 10.- Alloy Al-Cu (10 percent) chill-cast. Polishing Disa - 45 volts - 20 seconds. X400.

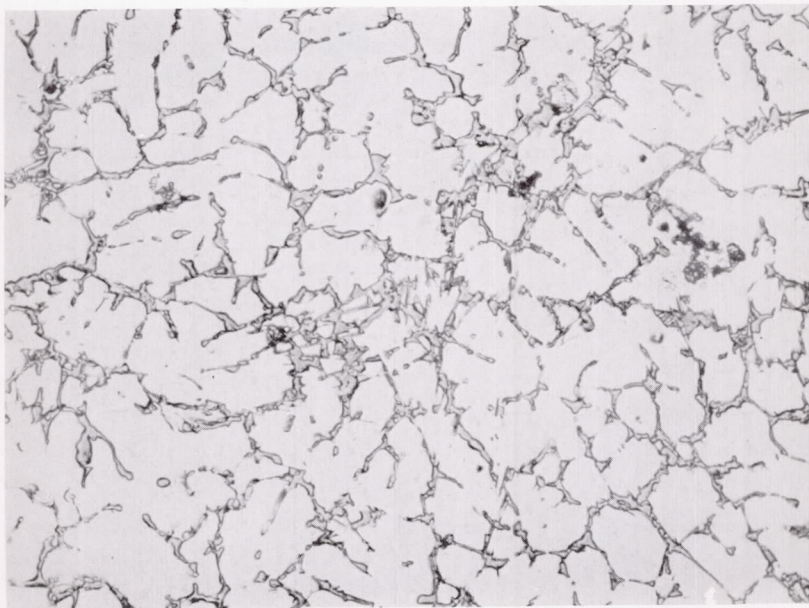


Figure 11.- Alloy Al-Cu (12 percent) chill-cast. Polishing Disa - 45 volts - 20 seconds. X160.

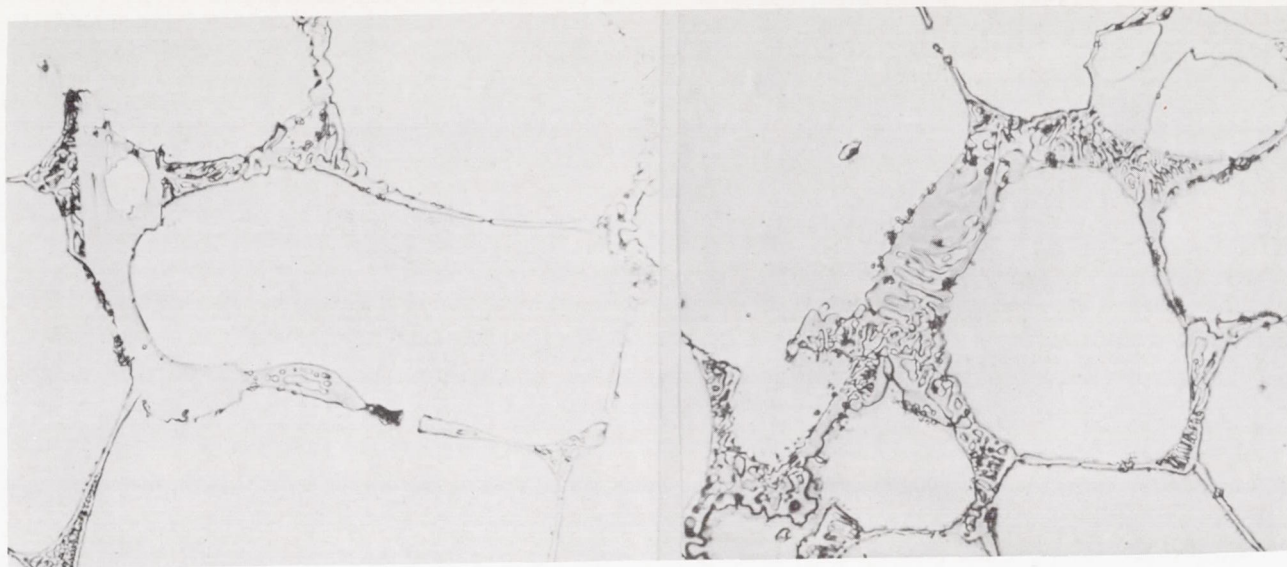


Figure 12.- Polishing Disa-Electropol 40 volts - 20 seconds. X630.

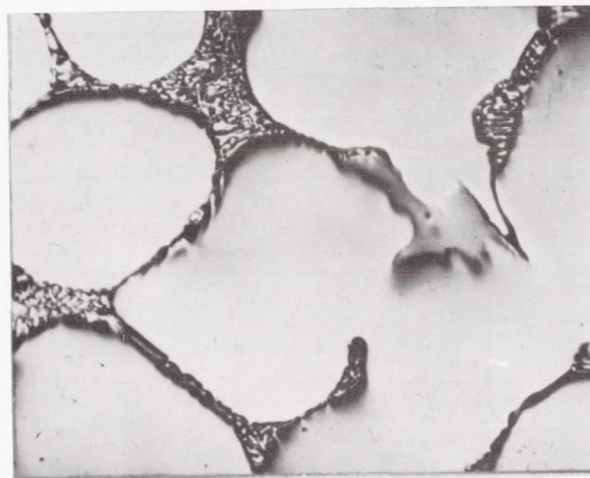


Figure 13.- Polishing in acetic-perchloric bath (one part perchloric acid - two parts acetic anhydride) 25 volts - 2 minutes. Note that the relief of the constituents is much more marked than on the same alloy when polished with the Disa. X600.

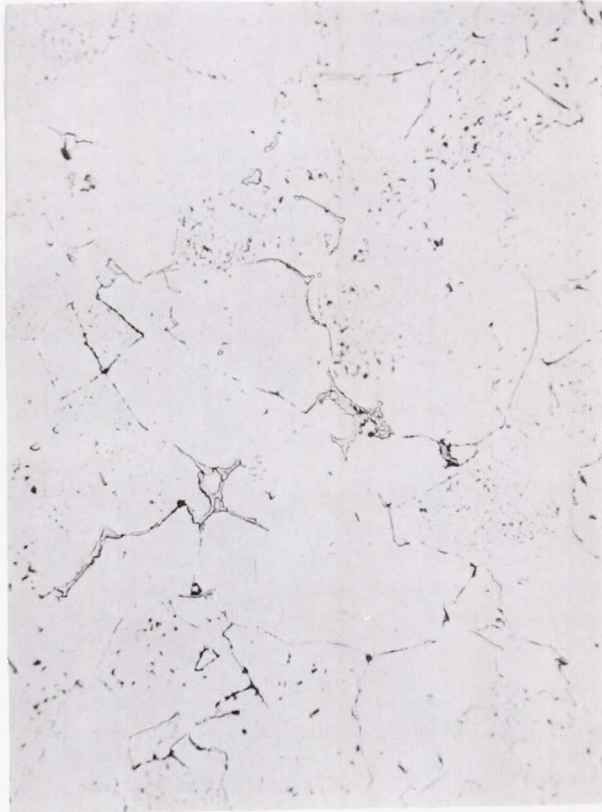


Figure 14.- Alloy aluminum-copper (4 percent) - magnesium (0.2-0.4 percent) - silicon (0.25 percent) - titanium (0.15 percent). Chill-cast state. X400.

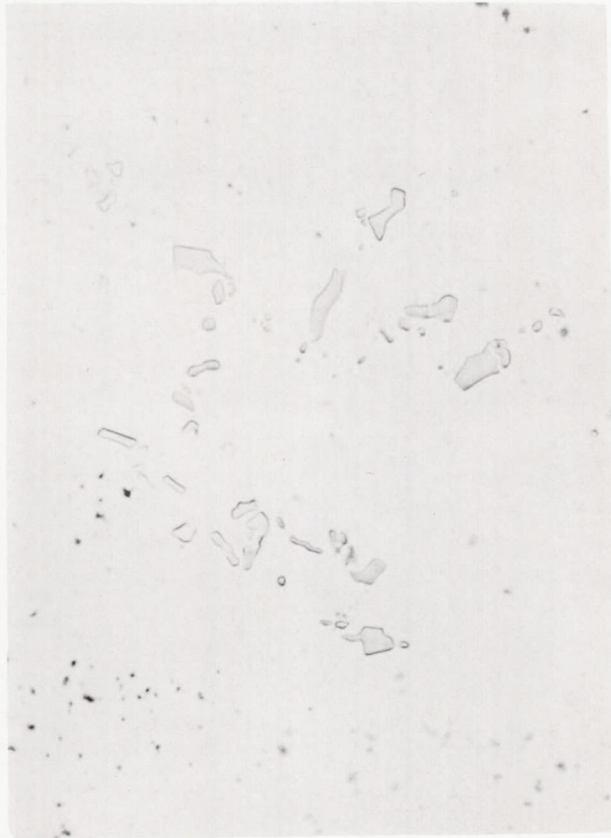


Figure 15.- Alloy "Fortal" aluminum-copper (4 percent) - magnesium (0.6 percent) - silicon (0.50 percent). Welded condition. X1000.

Alloy Y chill-cast aluminum-copper (4 percent) - nickel (2 percent) -
magnesium (1.5 percent) - silicon (0.5 percent).

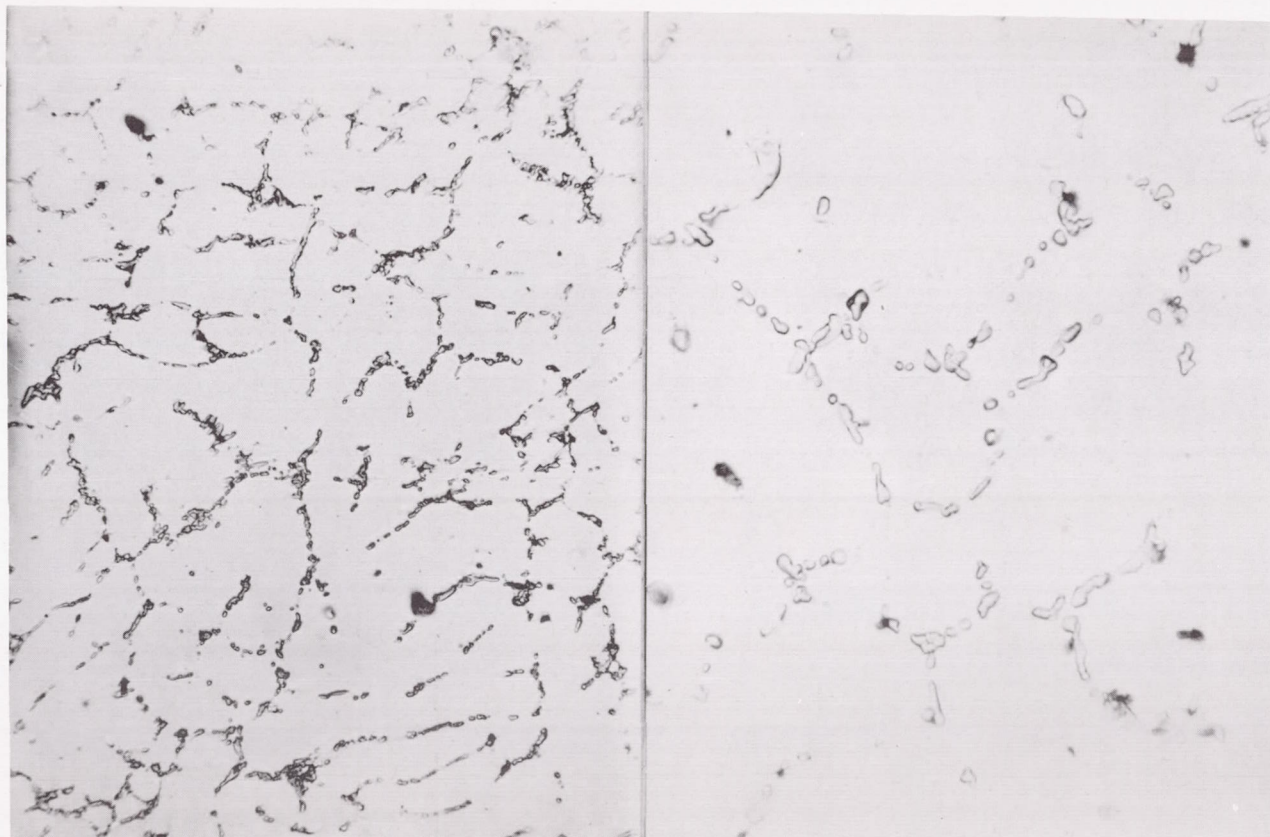


Figure 16.- X400.

Figure 17.- X1000.

Alloy aluminum-magnesium (7 percent).

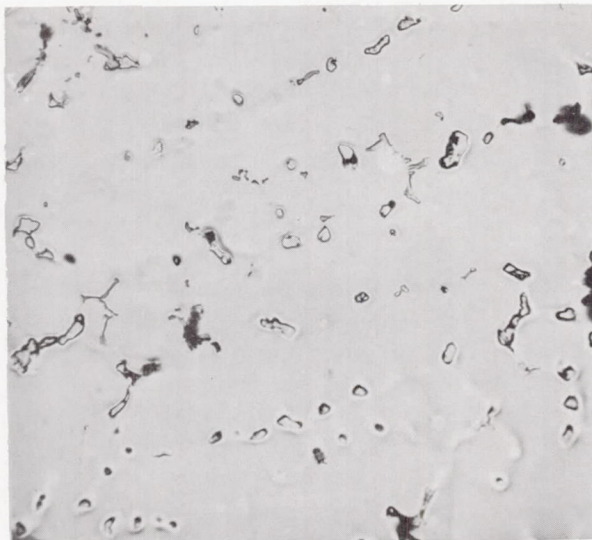


Figure 18.- Continuous billet rolled to 1/2. Section normal to the axis. Layout of the constituents. X160.

Alloy aluminum-magnesium (7 percent).

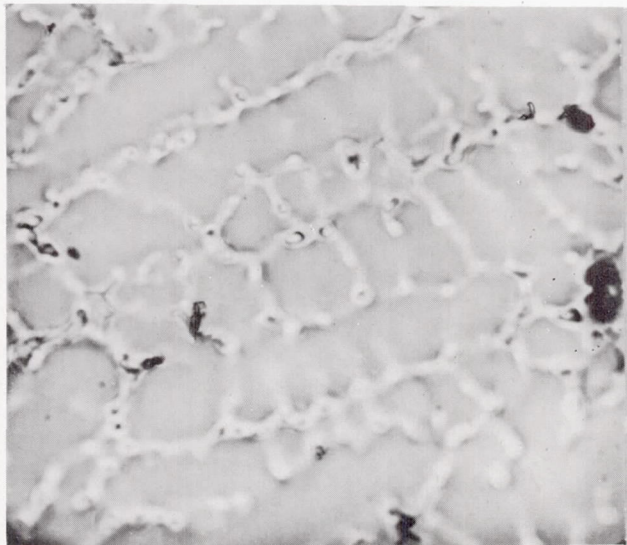


Figure 19.- Same specimen and same field as figure 18. Layout of the interdendritic pattern of heterogeneous solid solution containing the constituents. X160.

Alloy aluminum-magnesium (7 percent).

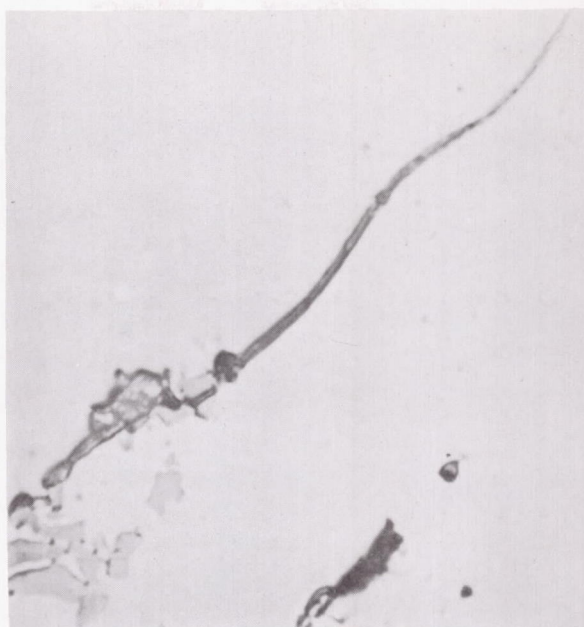


Figure 20.- Exterior surface of a cylindrical container obtained by hot-drawing and cold-forming of a disk taken from the billet figure 18. Intergranular fissure in relation to primary constituents (bottom, at right) and a precipitate (top, at left). X2000.

Alloy aluminum-magnesium (7 percent).

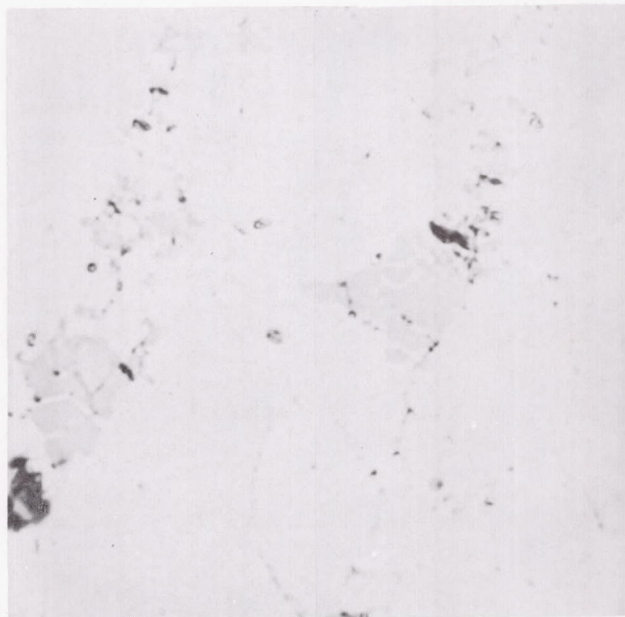


Figure 21.- Exterior surface of another container similar to the one in figure 20. Discontinuous attack on the grain boundaries (precipitate ?) and primary constituents. X2000.

Alloy aluminum-magnesium (7 percent).

Specimens taken from the bodies of various containers.

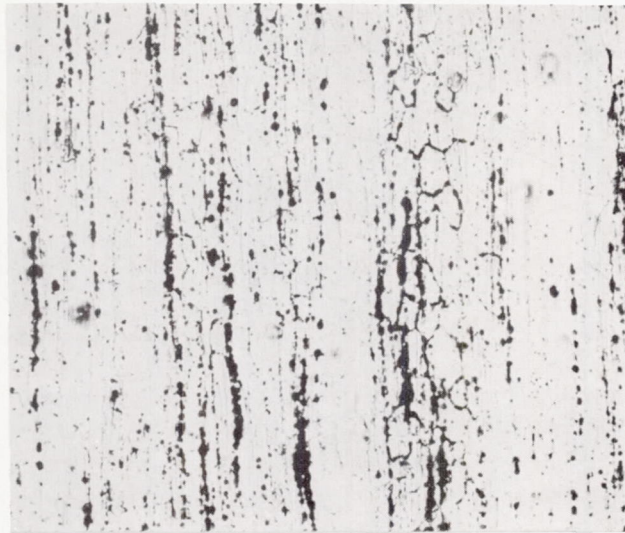


Figure 22.- Section of the wall, polished, then anodically oxidized for 10 minutes. Photograph taken in normal light. The network of grain boundaries is discontinuous. X160.

Alloy aluminum-magnesium (7 percent).

Specimens taken from the bodies of various containers.

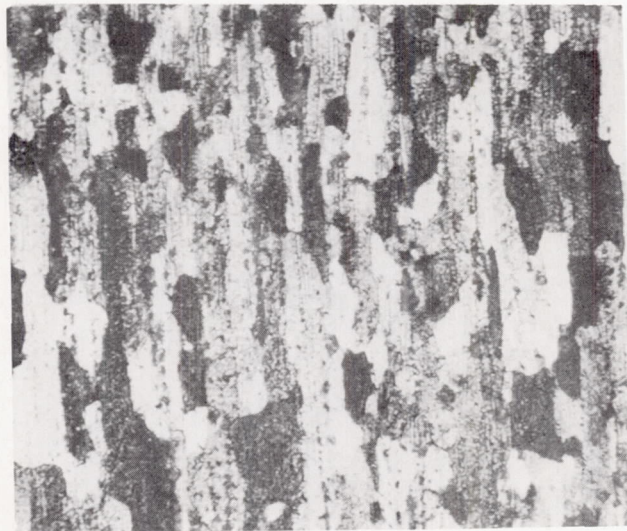


Figure 23.- Same specimen and same field as figure 22. Photograph taken in polarized light. The grains are revealed by the film of oxide. X160.

Alloy aluminum-magnesium (7 percent).

Specimens taken from the bodies of various containers.

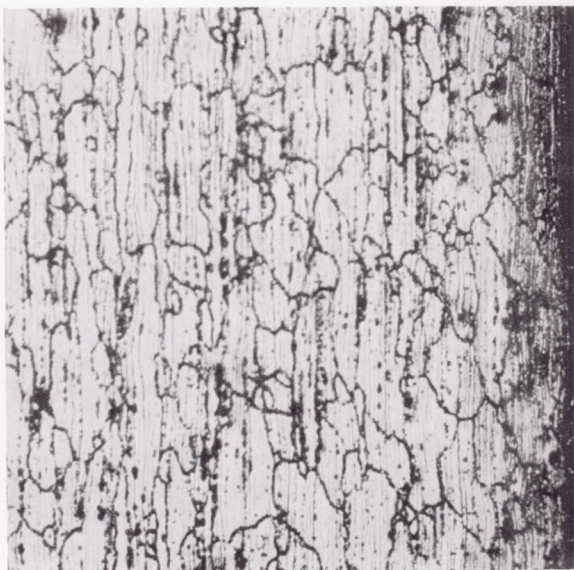


Figure 24.- Specimen from another container, treated under the same conditions as those in figure 22. Here the network of grain boundaries is very continuous. X160.

Alloy aluminum-magnesium (7 percent).

Specimens taken from the bodies of various containers.

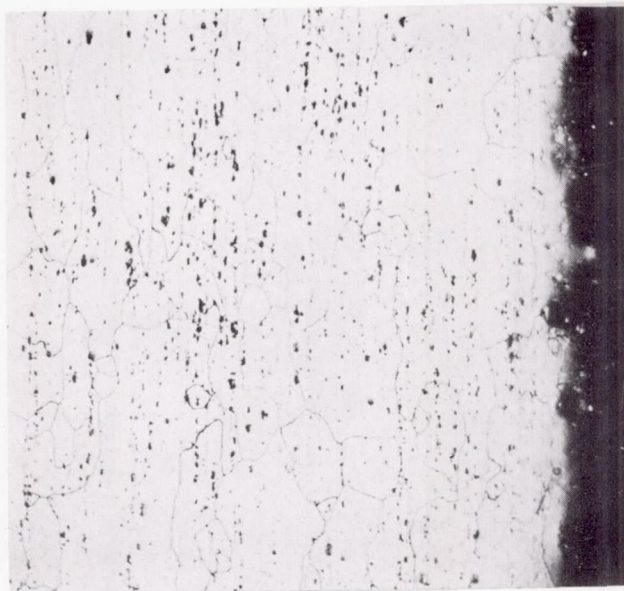


Figure 25.- Specimen from another container treated 16 hours at 160°. Attack 20 seconds in diluted HF. (With the same attack, the network of grain boundaries was invisible before the heat treatment.) X160.

Alloy aluminum-magnesium (7 percent).

Specimens taken from the bodies of various cylindric containers.



Figure 26.- Exterior surface. Specimen of figure 21. Attack Disa (method no. 2). The anodic oxidation or attack with diluted HF reveal equally the intergranular boundaries. X160.

Alloy aluminum-magnesium (7 percent).

Specimens taken from the bodies of various cylindric containers.



Figure 27.- Exterior surface. Specimen heated at 460°. Attack Disa (method no. 2). Neither anodic oxidation nor attack with diluted HF reveal the intergranular boundaries which are made evident by the Disa attack but appear less continuous than those of the specimen in figure 26. X160.

Alloy aluminum-magnesium (7 percent).
Specimens taken from the bodies of various cylindric containers.



Figure 28.- Section normal to the surfaces of the wall. Specimen of figure 26 - attack Disa (method no. 1). Slight etching of the grains without attack on their boundaries and without pits. X160.

Alloy aluminum-magnesium (7 percent).
Specimens taken from the bodies of various cylindric containers.

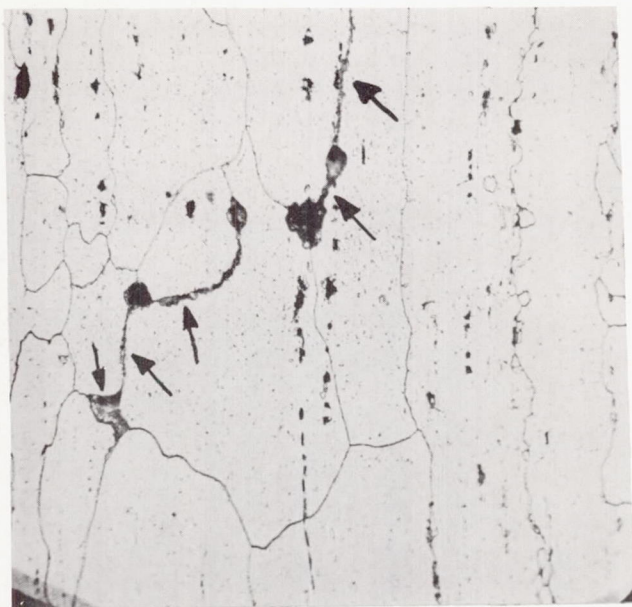


Figure 29.- Section of a specimen subjected to the mercury test, then heated 16 hours at 160° . Intergranular fissures (indicated by the arrows). X400.

Alloy refined "Alpax" (aluminum-silicon 13 percent) taken from the crucible immediately after refining.

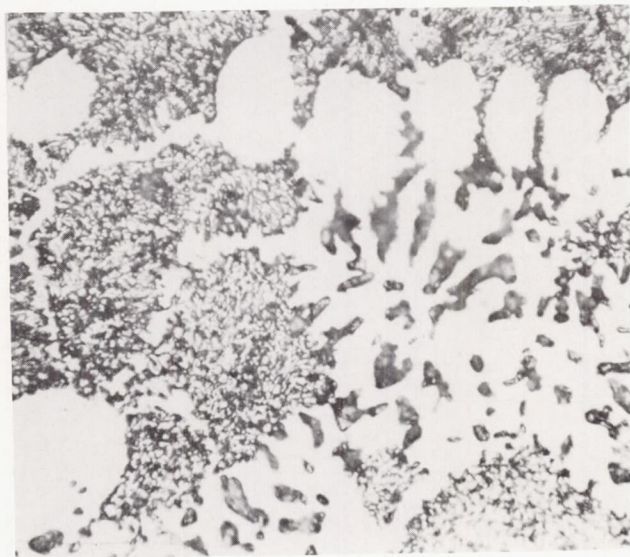


Figure 30.- Region of incomplete refining (remainder of large fragments of silicon). X400.

Alloy refined "Alpax" (aluminum-silicon 13 percent) taken from the crucible immediately after refining.



Figure 31.- Specimen of figure 30. Large fragments of silicon and constituents in small plates (Al_2Cu ?). X2000.

Alloy refined "Alpax" (aluminum-silicon 13 percent) taken from the crucible immediately after refining.

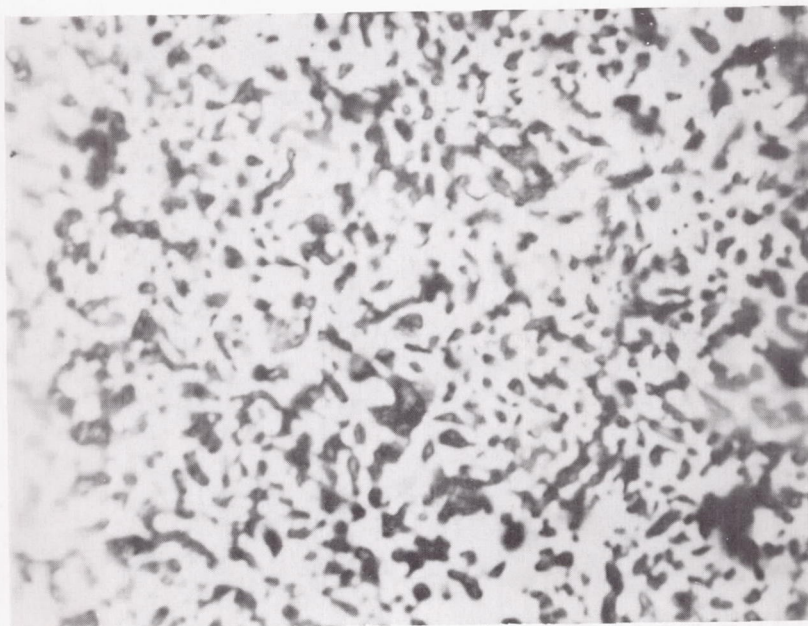


Figure 32.- Eutectic Al-Si in a region where the structure is better refined. X2000.

Aluminum-zinc-magnesium type alloys of high resistance.



Figure 33.- Alloy "Zicral" of the Aluminum Français (manufacture 1946 or 1947). Angle iron. Large constituents rich in chromium. X160.

Aluminum-zinc-magnesium type alloys of high resistance.



Figure 34.- Alloy T60 of the T.L.H. (manufacture 1944). Sheet metal plate. Polishing followed by attack Disa (method no. 1), leaving non-attacked the large constituents rich in chromium. X160.

Aluminum-zinc-magnesium type alloys of high resistance.

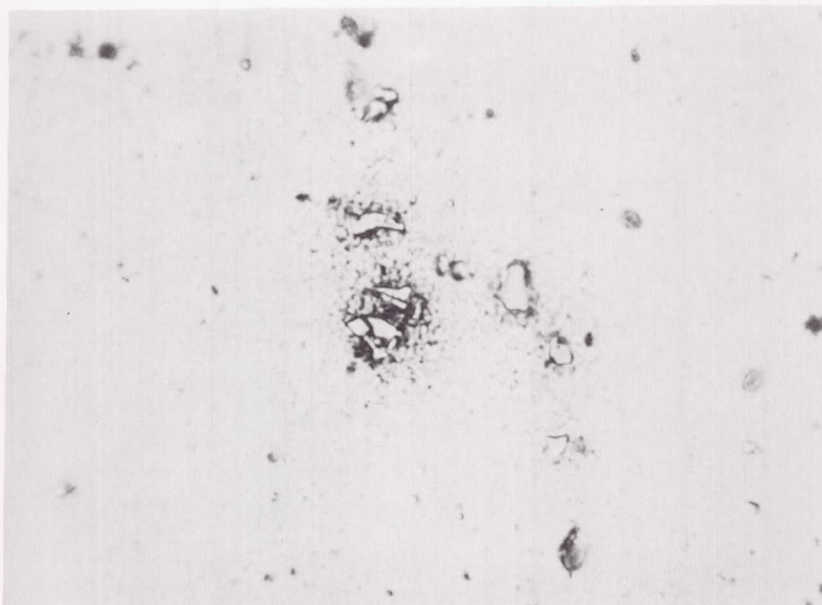


Figure 35.- American alloy 75ST - sheet - constituents rich in chromium much smaller and less numerous than in the alloys of figures 33 and 34. X1000.

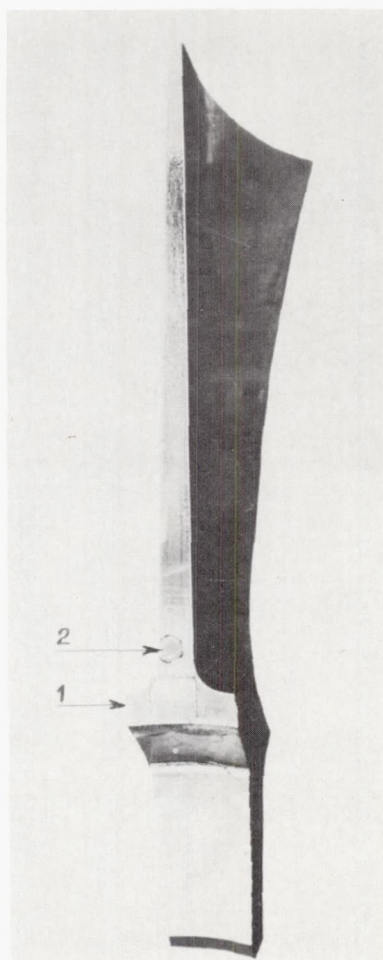


Figure 36.- Blade of forged and hot worked alloy RR 58 for the air compressor of the turboreactor Nene (manufacture Hispano Suiza). Examination of the plane section by local electrolytic polishing (1: square of 8-mm side; 2: circle of 5-mm diameter). X0.63.

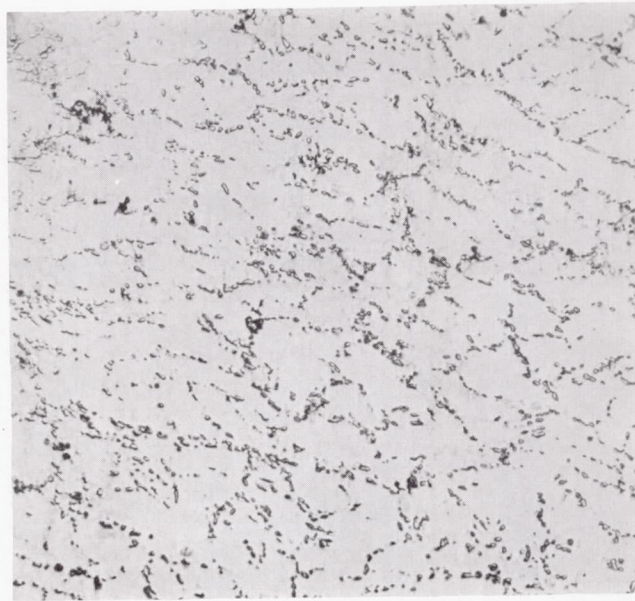


Figure 37.- Structure of the alloy on the region 1, figure 36. X160.

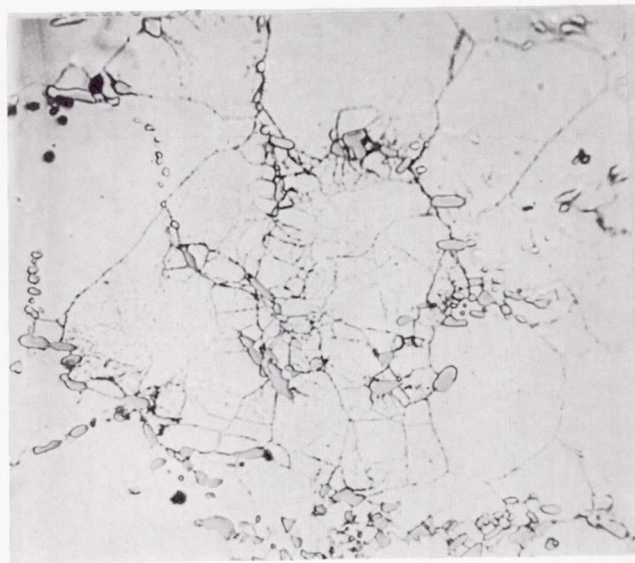


Figure 38.- Specimen of figure 37. Note the intergranular network containing precipitate and corresponding doubtlessly to polygonization boundaries. X630.

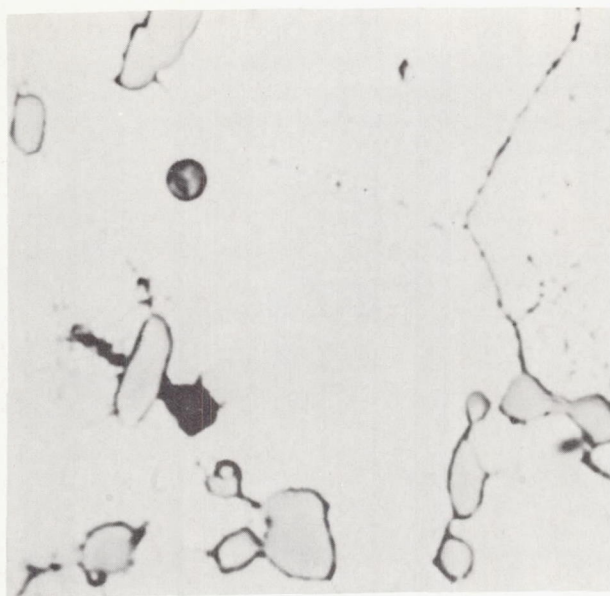


Figure 39.- Specimen of figure 37. Grain boundaries containing a more or less continuous precipitate. X2500.

Alloy "Vival" aluminum-magnesium (1 percent) - manganese (0.2 percent) - silicon (0.7 percent).

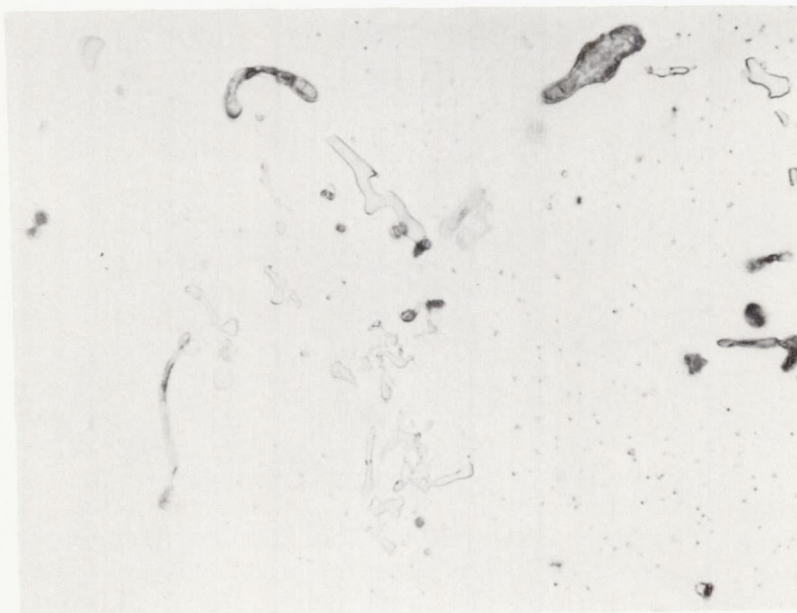


Figure 40.- Welded, annealed state. X1000.

Alloy "Vival" aluminum-magnesium (1 percent) - manganese (0.2 percent) - silicon (0.7 percent).

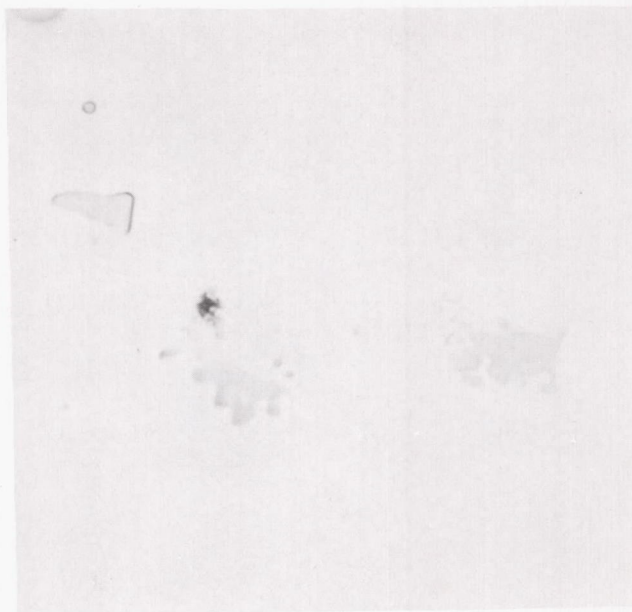


Figure 41.- Welded state. Heat treated 20 minutes at 550°, then 2 hours at 175°. Constituents in the process of dissolution. X2000.

Alloy "Vival" aluminum-magnesium (1 percent) - manganese
(0.2 percent) - silicon (0.7 percent).

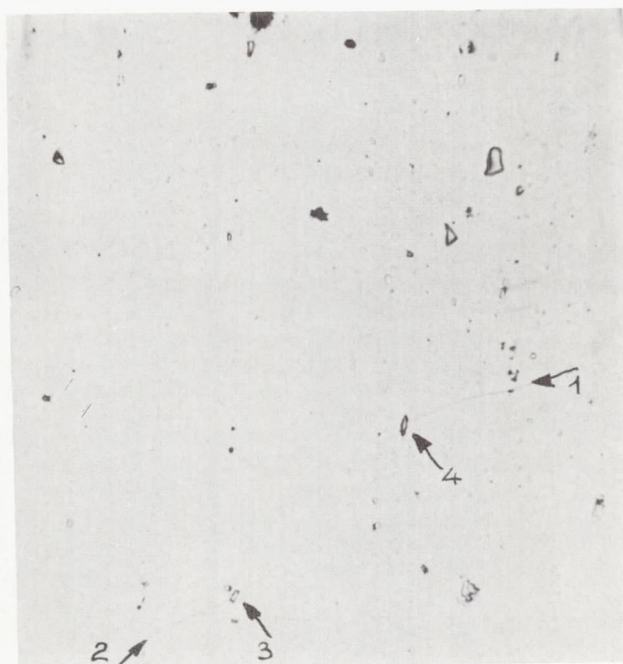


Figure 42.- Specimen of figure 41. Attack with diluted HF which makes the grain boundaries appear. One boundary is indicated by the arrows and 2. The arrows 3 and 4 indicate two small constituents on this boundary. X160.



Figure 43.- Alloy Al-Cu 4 percent, tempered 60 hours at 200° C.
Direct examination by electron microscope of a section reduced by
electrolytical polishing. X20,000.

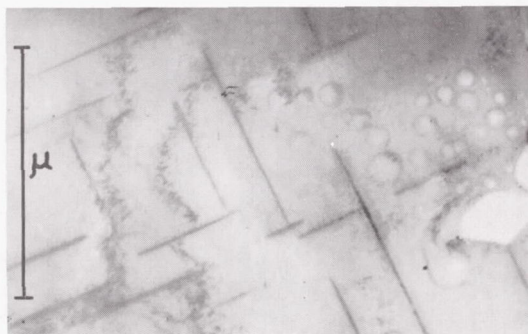


Figure 44.- Same alloy as in figure 43. Direct examination by electron
microscope of a section reduced by electrolytical polishing, then by
ion bombardment. X33,000.

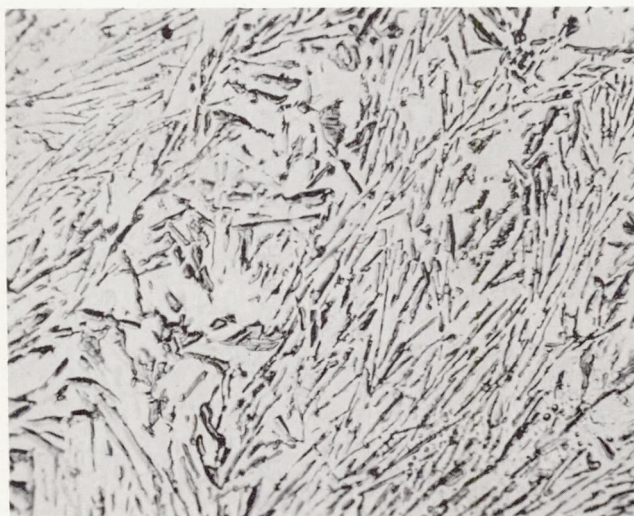


Figure 45.- Alloy containing 12.85 percent Si - 0.51 percent Fe - 0.38 percent Mn. Polishing on Disa Electropol with periodic inversion of the direction of the current (according to E. Knuth - Winterfeldt). X400.