TALAT Lecture 1202

Metallography of Aluminium alloys

20 pages, 7 Figures

Basic level

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Objectives

This lecture aims at providing a survey of the metallographic techniques available for the examination of aluminium and its alloys. The information must be sufficient to be sure that the students and the users are able to choose the most suitable technique to solve their problems in the examination of samples. The lecture should contain a direct understanding of the main problems in the metallography of the different classes of aluminium materials.

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1202.01. Introduction

The examination of microstructure is one of the principal means of evaluating alloys and products to determine the effects of various fabrication and thermal treatments and to analyse the cause of failure. Main microstructural changes occur during freezing, homogenisation, hot or cold working, annealing, etc. Good interpretation of the structure relies on having a complete history of the specimen.

In general, the metallography of aluminium and its alloys is a hard job in the meaning that aluminium alloys represent a great variety of chemical compositions and thus a wide range of hardnesses and different mechanical properties. Therefore the techniques required for metallographic examination may vary considerably between soft and hard alloys. Moreover, one specific alloy can contain several microstructural features, like matrix, second phases, dispersoids, grains, subgrains and thus grain boundaries or subboundaries according to the type of the alloy and its thermal or thermomechanical history. However, some methods of sample preparation and observation are quite general and apply to all aluminium alloys. In other cases one should refer to specific developed methods.

As a general rule, examination should start at normal eye vision level and proceed to higher magnification. Simplicity and cost make optical examination (macro and micro) the most useful. When the magnification and the depth of focus become too low, the electron microscopies are required.

1202.02 Sample preparation

Aluminium alloys require the same principle of preparation for examination as most metals. Careful visual inspection of the part to be examined should precede cutting or etching. Fracture surfaces must be carefully preserved against abrasion or contamination. If the part is difficult to handle and has to be sectioned, care should be taken to cut the material along directions determined by the working process and by other interesting criteria. As an example, if the alloy has been rolled, it can be interesting to examine the evolution of microstructure along the rolling direction and so the part must be cut in the same direction.

1202.02.01 Optical microscopy: mechanical grinding, mechanical polishing, etching, anodising

The general **metallographic sample preparation for optical microscopy** includes a series of steps described in the following paragraph. The selected part of the material is cut by a SiC abrasive saw at a certain distance from the plane to be observed, because a thickness of several tens of microns will be then removed by mechanical grinding. A lubricant for cutting is used to avoid temperature increases and structure modification of the specimen. The sectioning is usually followed by mounting of the sample in a plastic medium to form a cylindrical piece that can be handled during grinding and polishing. This stage is not necessary if the sample is large enough. Cold mounting has to be preferred to the hot one if the alloy is sensitive to microstructural modifications (precipitation) also at low temperature (around 100°C).

Mechanical grinding is performed in successive steps using SiC abrasive papers of different grit sizes, usually 180, 220, 320, 600, 800, 1000, 1200, 2400 grit. The starting grit depends on the type of cut surface to be removed. The abrasive particles embed easily into soft aluminium alloys. So a wet grinding (water) to flush away these particles and a small pressure on the specimen are recommended.

The following step is **the mechanical polishing**. It is important for removing the surface scratches produced during grinding. The mechanical polishing is usually performed in two steps : rough and final polishing. The rough polishing is performed using 3 and 1 µm diamond paste on a short nap cloth disk. The lubricant can be a solution of alcohol and propylene glycol. The final step is made by 0.25 µm diamond paste. It is important to wash the sample after every step in an ultrasonic bath to remove all the abrasive. In cases where polishing with 1 or ¼ µm diamond paste does not produce a sufficiently deformation - free and scratch-free, highly reflective surface, as it is the case of pure aluminium and its soft alloys, the final polishing can be carried out using metal oxides in aqueous suspension. The most commonly used oxides are colloidal silica (SiO_2) and alumina (Al_2O_3). Silica comes as a ready made colloidal suspension OP-S 0.04 µm. The advantages of silica over alumina are the particle size distribution which is much narrower than in alumina and the particles which do not agglomerate and are always in suspension. This means that there is less chance of fine scratches and it is easy to apply because it does not need continuous stirring. The cloths used for final polishing are usually soft compared to the harder cloth for diamond polishing.

The polishing can be also performed in an electrolytic way. This technique is suited for polishing homogeneous structure such as pure aluminium or very soft alloys which are difficult to polish mechanically [1,2]. Ref. 1 reports some of the electropolishing solutions frequently used for aluminium alloys. The principle of **electrolytic polishing** is described in the section which covers sample preparation for transmission electron microscopy (TEM). After polishing, the sample is ready for the last step before observation; this is called the etching. **Etching** is basically a controlled corrosion process resulting from electrolytic action between surface areas of different potential. With pure metals and single-phase alloys, a potential is produced between differently oriented grains, between grain boundaries and grain interiors, between impurity phases and the matrix or at concentration gradients in the single-phase alloys. With two phase or multiphase alloys, potential differences are also present between phases of different composition. These potential differences are used to produce controlled dissolution. The quality of the polishing influences the development of the true microstructure. A faulty preparation can lead to misinterpretation of the structure. In general wiping of the surface with moist cotton under running water is adequate, although ultrasonic cleaning especially if cracks or pores are present, is preferable [3].

The etchants for use in microscopic examination of aluminium alloys are numerous and are illustrated in several books [2,3,4]. The most common for practical use are the followings :

- **Keller's reagent** - 2ml HF (48%) + 3ml HCl + 5ml HNO_3 + 190ml H_2O. This etchant gives the possibility to reveal grain boundary contrast and precipitates in several wrought alloys.
- **1g NaOH + 100ml. H_2O** - This solution is useful for grain boundary contrast in 6xxx series.
- **(HF etch)** - 1 ml (HF) (48%) + 200ml H_2O. It is used for constituent identification in cast alloys, especially those containing Si.

It is clear for example that the grain structure can not be easily revealed in every alloy. On a specimen with low alloying metal, the etching produces steps at the grain boundaries which do not provide good contrast. In this case anodising is preferable, as described below.

Anodising or anodic oxidation is an electrolytic etching process for depositing an oxide film on the metal surface that is often epitaxial to the underlying grain structure. The resulting interference colours are a function of the anodic film thickness, which depends on the anodising voltage, the solution and the composition and/or structure present in the specimen [2,5]. Anodising was first applied to the study of the grain structure of aluminium. Although aluminium has a cubic crystal structure and is therefore isotropic, the oxide film produced permits the grain structure to be observed under crossed-polarised illumination. If the film is thin, interference colours are produced and physical and chemical inhomogeneities can be observed. Generally thicker films are produced and the optical effect is ascribed to the surface structure of the oxide film or to submicroscopic profile of the interface of the metal and oxide [3]. The solution used for anodising aluminium and its alloys is called **Barker's reagent** - 5ml HBF_4 (48%) + 200ml. H_2O (anodising conditions : $J=0.2\ A/cm^2$ for 40-80 s at room temperature). Actually the anodising voltage can be selected by trial and error. It is recommended to electropolish the sample before anodising.

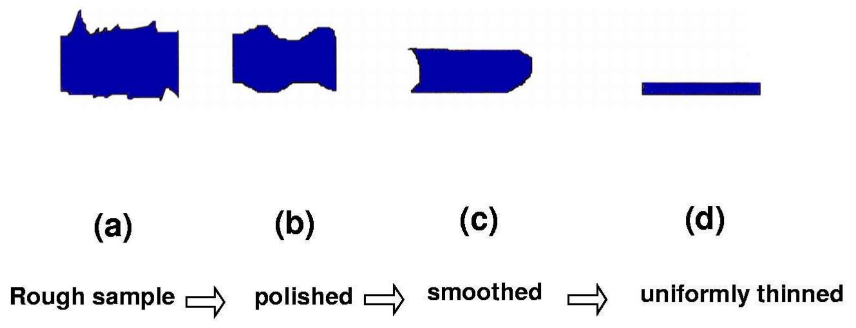
In other materials, grain boundary precipitation can delineate the grain boundary itself if of course the alloy is heat treatable. A similar thinking concerns the examination of precipitates by optical microscopy. Moreover, the identification of precipitates requires other techniques like x-ray analysis or electron microscopy.

1202.02.02 SEM and TEM : electropolishing, dimpling, ion milling

Specimen preparation for electron microscopy has to be differentiated between scanning and transmission electron microscopy. In general, for the scanning electron microscope (SEM) the sample preparation is similar to the one for optical microscopy. The different results in observations are eventually attributed to the technique that is based on the interaction between electrons and metals and not on visible light. If the interesting area is a fracture surface, no special sample preparation is required, only a careful cleaning of the surface to be examined.

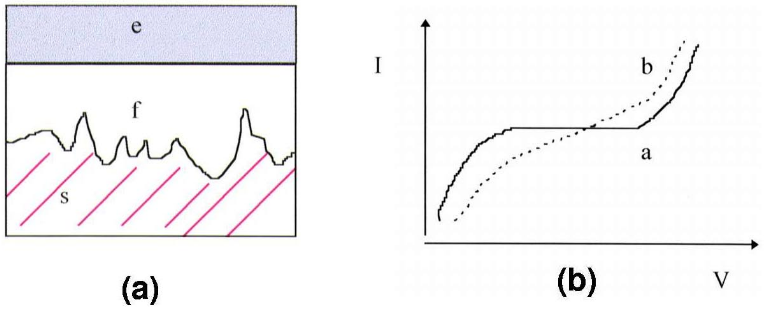
For transmission electron microscopy (TEM), sample preparation is more difficult compared with SEM and optical microscopy, especially because of the very thin and small pieces that have to be handled. The procedure for TEM preparation of aluminium and its alloys is standard, and like that used for other metallic materials [1]. Extreme care must be taken to avoid the introduction of artificial defects into the microstructure, especially during cutting and mechanical grinding, that can substantially modify its evaluation. All the preparation steps are listed in the following section but more emphasis will be given to the final thinning techniques. First the sample is cut in slice 1 mm thick by SiC abrasive saws and then mechanically ground and polished down to ≈ 100 µm using the same papers and clothes as for optical microscopy preparation. In this case the foil has to be polished on both sides. When the thickness of the slice has reached 100µm, other techniques are used to thin the sheet or disc specimen down to its final electron-transparent thickness. These are the electropolishing or the dimpling and the ion milling. These three techniques are described hereafter.

The principle of **electropolishing** is quite simple. An electrolytic cell is established with the specimen as the anode and an appropriate potential is applied so that the sample is dissolved in a controlled way (**Figure 1202.02.01**). Electropolishing is performed until a hole has formed : this means that the region around the hole should be thin enough for TEM. Experimentally there are many variables to control like cell geometry, applied potential, temperature and stirring velocity of the solution. The electropolishing must remove very fine surface irregularities and must thin the specimen uniformly.



TALAT Training in Aluminium Application Technologies | The stages of electropolishing | 1202.02.01

The electrolyte must contain an oxidising agent together with reagent that will form a thin but stable viscous film. The fine electropolishing action is achieved by dissolution controlled by the length of the diffusion path through the viscous film to the electrolyte (**Figure 1202.02.02**).



(a)

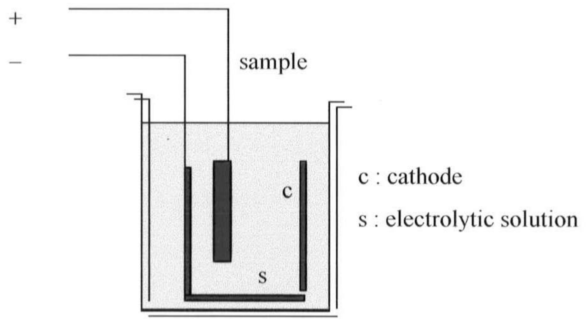
(b)

(a) A viscous film (f) forms between the sample (s) and the electrolyte (e); high spots have a shorter diffusion path through the layer and polish faster.

(b) Current-voltage curves for electropolishing; curve a is ideal with the best polishing conditions lying in the central plateau, but more commonly a curve such as b is found [1].

TALAT Training in Aluminium Application Technologies | **The mechanism of electropolishing** | **1202.02.02**

In order to start polishing, the essential components are the electrolyte, the cell and a power supply (usually d.c.) to provide 5-50 V (see **Figure 1202.02.03**).



Schematic representation of the electropolishing technique

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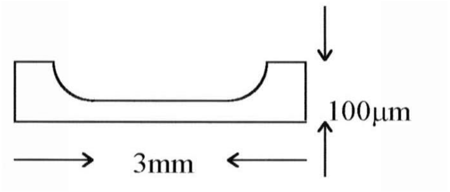
Some of the most used electrolytic solutions are listed in ref. 1.

In practice, after the slice is uniformly 50µm in thickness, it is punched into several disc of three mm in diameter and then every disc is again electropolished in an automatic jet polisher. In a jet polisher a jet of electrolyte is directed at the centre of the disc on both sides of the specimen until it is appreciably thinned. This produces an ideal specimen for mounting in the TEM with strong edges that support the thin areas in the centre. Usually the electrolyte and the other parameters are the same as for electropolishing.

If it is desirable to have a disc with a depression in the centre for the final thinning (for example when using the ion milling technique) it is necessary to **mechanically dimple** the disc. This is a means by which a disc is created with a depression in its centre only on one side. Usually a 1mm thickness metal wheel tool is all that required to dimple discs cut by spark machining or ultrasonic drilling. During dimpling, rough abrasive diamond paste is used for grinding and fine diamond paste for polishing with felt wheels. Care must be taken in all cases that the thickness remaining in the centre of the disc is not so small that damage has occurred through the final thin area. If the starting disc thickness is around 100µm, the depression can reach 2/3 of its initial thickness (**Figure 1202.02.04**).



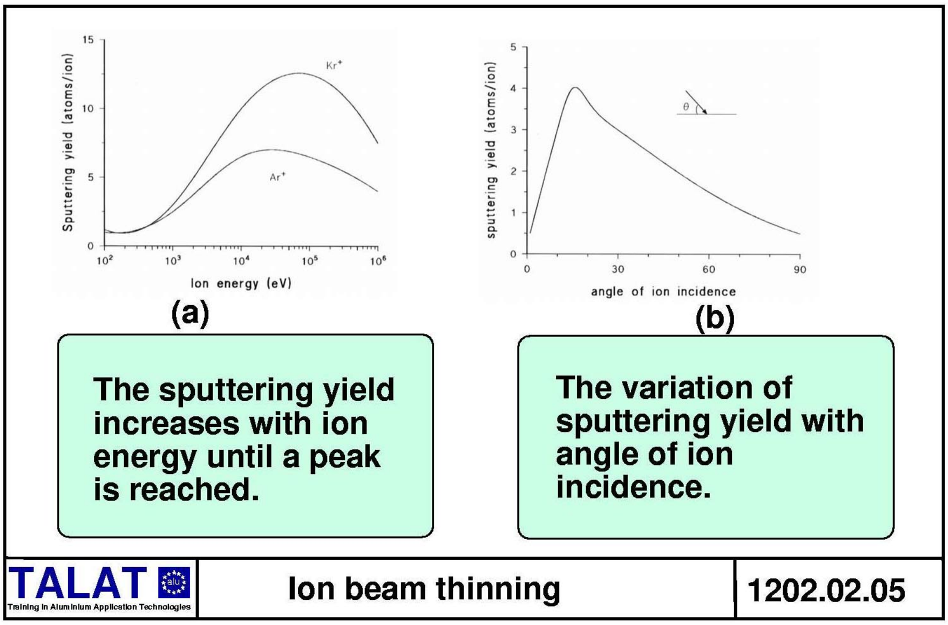
The profile of a dimpled specimen

TALAT Training in Aluminium Application Technologies | **Disc specimen for TEM** | **1202.02.04**

If the material is composed of two phases and one phase electropolishes faster than the other, it is more suitable to use **ion milling** for the final thinning. In aluminium alloys, ion milling is useful for Al-Si system and their metal matrix composites.

The principle of ion beam thinning is rather simple. A beam of inert gas ions or atoms is directed at the disc specimen from which it removes surface atoms in a process known as sputtering. If this can be achieved without the creation of artefactual damage then the ion beam thinning is an ideal method for the preparation of foils from both conducting and non-conducting materials. However it is necessary to control several undesirable effects like ion implantation, the development of a rough surface and the heating of the specimen that for aluminium alloys is a very important problem to avoid. For these reasons it is necessary to control the nature of ions, their energy and direction of incidence and their frequency of arrival (ion beam current). Sputtering occurs when any ion carrying more than about 100eV of energy hits a surface. The number of atoms ejected by each incident ion is called the sputtering yield Y. In general Y increases with ion energy and ion mass, but decreases with increasing specimen atom mass. The argon is the inert gas mostly used for ion beam thinning because of its reasonable cost and good sputtering yield. The optimal ion energy is easy to select according to **Figure**

1202.02.05 (a), where the Y at high ion energy decreases because the ion start to implant in the surface instead of ejecting atoms.



(a)

(b)

The sputtering yield increases with ion energy until a peak is reached.

The variation of sputtering yield with angle of ion incidence.

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Ion beam thinning

1202.02.05

The value of energy corresponds to an accelerating voltage of 4-6 kV. The sputtering yields depend also on incident angle as shown in **Figure 1202.02.05** (b). Usually the starting incident angle is around 14° to increase the yield, but after a while it is decreased to 12° to a have a better control on the thinning. For aluminium alloys it is recommended to have a cooled specimen holder because the sample temperature can increase also to 100°C or more due to ion interactions with the material and this can affect the structure.

The ion beam technique has become useful also for ceramics and non-conducting materials. It is less useful for light ductile conducting metals where the microstructural damage introduced by ion implanting in the surface may lead to confusing artefacts.

1202.03 The techniques used in metallography of aluminium and its alloys

This section will deal with the main techniques used in optical and electron microscopy for observation of aluminium and its alloys. As the techniques are common to other metallic materials, tables will be reported containing a list of the most common observation techniques with emphasis on the visible structure and the sample preparation required. Information about specific techniques can be found in the indicated references.

1202.03.01 Polarised light, interference contrast [2]

Light microscopy is the major tool for microstructural examination of aluminium alloys and is recommended for use before electron microscopy. It can reach magnification of 1500X and resolves features as small as 0.1µm. Usually, optical microscopy can show the size and distribution of resolvable particles, and shows the state of the crystal structure. It does not reveal precipitate particles responsible for precipitation hardening or dislocation arrangement. Generally, analysis of these conditions is the domain of electron microscopy.

Table I reports some of the most common techniques used in optical microscopy for observation of metallic materials. In the following a description of polarised light and interference contrast mode will be reported with reference to their use for aluminium alloys.

Table I : optical microscopy observation techniques

technique	*sample preparation*	*structure*
bright field	normal	g, gb, p
dark field	normal	p, gb
polarised light	anodisation	g
interference contrast	normal	g, gb, p

g : grain, gb : grain boundary, p : phases and precipitates, fs : fracture surface, d : defects and dislocations, i : interfaces, e : etched, u : unetched

The polarised light. An isotropic metal (cubic or amorphous crystal structure) transmits or reflects light of the same velocity in all directions. If a plane polariser light beam strikes normal to the surface of an isotropic metal, it will be reflected as a plane-polarised beam with the same azimuth of polarisation. The amplitude of the beam of course will be reduced by an amount that varies with the reflectivity of the particular metal. If a plane-polarised light beam is passed through a second polarising filter (the analyser) placed at 90°C to the polariser, the light will be extinguished. This position of the polariser and analyser is referred as crossed. If an anisotropic metal is substituted for an isotropic metal in the previous experiment, an image of the microstructure will be observed. The rotation of the sample under the beam of 360° produces four positions of maximum and minimum light intensity in each grain. The use of monochromatic light is recommended, but white light also produces colour contrast effects [2]. A strain free, clean surface is required for best results. So electropolishing is the best preparation method. The technique of anodising, frequently used with aluminium, produces a thick oxide film on the metal surface electrolytically : irregularities in the film lead to double reflection. Although the polarising response of anodised specimens has been attributed to optical anisotropy of the film, experimentation has shown that the effect is due to film surface irregularities. In the case of aluminium, this technique is primarily used to reveal the grain structure but not the determination of crystallographic orientation of the grains.

Interference-contrast technique. This mode is useful for studying microstructures which exhibit little or no intensity contrast. It produces images with emphasised topographic details similar to those observed using oblique illumination. A detail that is invisible in bright field like small precipitate or grain boundary may be revealed with interference contrast illumination. Usually this is achieved using the Normansky-modified Wollaston prism [7]. When light passes through the double-quartz prism, it is split in two waves front with a path difference : that is one wave front is slightly ahead of the other. When this light is reflected from the specimen surface, the path difference changes due to the height differences on the surface. At the end, the image intensity in the field of view is a function of geometric height differences and phase jumps. Therefore the intensity differences produces relief effects resembling unilateral, oblique illumination [2].

1202.03.02 Electron channelling contrast and electron channelling patterns

The SEM is mainly used for the examination of thick samples. Electrons which are emitted or backscattered from the specimen are collected to provide topographical information (secondary electrons), atomic number or orientation information if backscattered electrons are used. In addition other signals like electron beam induced current and light cathodoluminescence can be collected.

Table II reports some techniques used in scanning electron microscopy for observation of metallic materials. In this chapter a description of electron channelling contrast and electron channelling pattern (ecc and ecp) modes will be reported with reference to their use for aluminium alloys. This choice has been attributed to the increasing industrial importance of both techniques for understanding the recovery and the recrystallisation processes. The ecc and ecp represent very powerful techniques to achieve this.

Table II : observation techniques for SEM

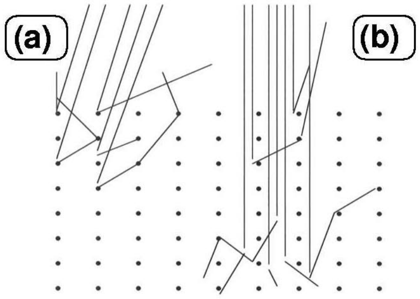
technique	*sample preparation*	*structure*
secondary electron images	normal	g, gb, p, fs
back scattered electron images	normal, e, u	g , gb, p
channelling images	electropolishing	g , gb, p
chemical analysis	normal	-
local orientation measurements (ebsp)	electropolishing	-

g : grain, gb : grain boundary, p : phases and precipitates, fs : fracture surface,
d : defects and dislocations, i : interfaces, e : etched, u : unetched

The study of crystalline materials in the SEM has been greatly advantaged by the discovery of electron channelling effects from bulk crystals [7]. The **electron-channelling pattern** can provide crystal orientation from a surface layer less than 50nm thick. Electron channelling contrast can be employed in images scanned in the conventional manner to provide images of grains and other crystallographic features.

The interaction of the primary electrons of the beam with the atoms of the solid results in deflections of the electron trajectories from the incident direction due to high angle elastic scattering and a loss of energy due to inelastic scattering. In a crystalline solid the periodicity of the atom arrangement can affect the way in which the primary beam interact. The electron channelling effect arises because of the different atom packing density observed along the crystallographic directions. When the angle between the incident beam and one of the crystal axes decreases (see **Figure 1202.03.01**), the electrons penetrate more deeply into the crystal passing along the channels of atoms. The chance of escaping for the incident electrons decreases near exponentially with depth below the surface. So the electrons that channel into the crystal have a lower probability to escape from the crystal respect to ones close to the surface. This difference creates the contrast. Actually, in order for the ecc effect to produce contrast in a SEM image the condition $\theta < \theta_B$ and $\theta > \theta_B$ must be satisfied at sequential points in the SEM images. If these conditions are satisfied for a particular set of planes contrast will occur becuase of differences from point to point in the number of emitted electrons (θ is the incident angle and θ_B the Bragg angle). Electron channeling contrast is thus a form of pure emission number contrast.

In practice this means that the channelling mode will be achieved only with the incident rays near parallel to the optical axis.



Simple model of electronchannelling effect

The beam incidence angle at (a) causes strong interaction at the surface. At (b) the angle results in electron penetration in channels through many atom layers before interaction [8].

TALAT Training in Aluminium Application Technologies | **Electronchannelling** | **1202.03.01**

If a complete two-dimensional raster is scanned on the crystal, as would normally be done in the SEM, then other sets of lattice planes will also be able to contribute to the contrast and the resultant signal plot, the **electron channelling pattern**, will show bands of contrast from all sets of planes normal or nearly normal to the surface. The width of each band will be twice the appropriate Bragg angle for the set of lattice planes from which it comes and the angle between the bands will be the angle between the corresponding sets of planes. This ecp thus has the symmetry of the crystal lattice in the area examined. If the crystal is tilted or rotated the bands will move as if fixed rigidly to the lattice. Any of the electron signals from the specimen (backscattered, secondary, specimens current etc.) will show equivalent contrast features and whichever is the most convenient can be used [8]. The ways in which the technique can operated are well illustrated in ref. 8,9,10,11. The sample to be examined by this technique is usually electropolished to obtain a strain free image and a clear pattern.

1202.03.03 High resolution electron microscopy (HREM) and high voltage electron microscopy (HVEM)

Many of the structures observed by light microscopy can be studied in more detail in the electron microscope. Furthermore, information that can not be obtained by optical microscopy become available. Thus in addition to grain structure of the material, subgrains and dislocation structure can be observed and also crystallographic orientation can be measured.

As far as the TEM observation techniques are concerned, it is not the author's intention to describe the several modes of TEM observation because the subject is very wide and complex and the bibliography to refer to is exhaustive. Table III lists common techniques of observation with references to the type of structural information obtainable. In the following, a short comment is dedicated to two powerful techniques : high voltage and high resolution electron microscopy (HVEM and HREM).

Table III : TEM observation techniques

technique	***sample preparation***	***structure***
bright field	normal	g, gb, p, d
dark field	normal	p, d
sadp	normal	-
HREM	normal	p, i
HVEM	normal	p, d
CBED	normal	-

g : grain, gb : grain boundary, p : phases and precipitates, fs : fracture surface, d : defects and dislocations, i : interfaces, e : etched, u : unetched

Most commercial microscopes operate between 100 and 200kV but a significant number of high voltage electron microscopes (hvem) can reach an accelerating voltage of 1MV. A major difference is in their physical size: HVEM requires much larger lenses and much more radiation shielding. The larger space available in HVEM has led naturally to the development of stages which allows in situ experiments to be carried out, like tensile stages and environmental stages. Tilting about two orthorhombic axes of ± 60° and ± 45° is a common requirements in diffraction analysis but if the microscope is designed for high resolution electron microscopy (HREM) then the amount of tilt available is reduced. Typically 1MV microscopes allow the examination of samples which are about five times the thickness of those which can be examined at 100kV. Loss of intensity occurs because the various scattering mechanisms combine to reduce the intensity of those electrons accepted by the objective aperture.

Even more sophisticated imaging and microanalytical techniques are being developed for studying aluminium structure in greater detail. High resolution TEM is capable of directly imaging the lattice structure, contributing to the understanding of lattice defects and the early stages of precipitate formation in some alloy systems.

HREM leads to some differences from conventional diffraction contrast microscopy. In HREM the high angle information must be allowed to interfere in the image plane in order to resolve the periodicity of the object, whereas in TEM diffraction contrast only one axial beam is allowed through the objective aperture. Thus an objective lens of very small spherical aberration is required in HREM so that the high angle information retains the correct phase relationship with respect to low angle information so that when they interfere in the image plane they reproduce faithfully the object periodicity. Additionally coherent illumination is required in order to obtain the correct phase relationships [12].

1202.04 The metallography of the different class of aluminium alloy

Aluminium and its alloys are divided in two categories, cast and wrought and each of this group is further subdivided into classes according to the composition. Table IV reports the classes with the main alloying element.

Table IV : classes of aluminium alloys

Cast alloys		**Wrought alloys**	
1xx.x	**aluminium ≥ 99.00%**	**1xxx**	**aluminium ≥ 99.00%**
2xx.x	**copper**	**2xxx**	**copper**
3xx.x	**silicon (with Cu and Mg)**	**3xxx**	**manganese**
4xx.x	**silicon**	**4xxx**	**silicon**
5xx.x	**magnesium**	**5xxx**	**magnesium**
7xx.x	**zinc**	**6xxx**	**magnesium and silicon**
8xx.x	**tin**	**7xx.x**	**zinc**
9xx.x	**other element**	**8xxx**	**other element**

Experience suggests that these alloys are suited to conventional metallographic preparation techniques and observations. The goal is the understanding of the appropriate technique for the observation of a certain class of alloys according to their processing history.

1202.04.01 Commercial purity of aluminium

The structure of commercial pure aluminium can derive from a cast process or from a wrought process. One of the problems is that the contrast is not relevant during observation by optical microscopy because of the lack of constituents and alloying element (cf. figs. 1 and 2 in ref. 3). This is a typical example where the anodising can help a lot for the grain structure observation of the aluminium, as illustrated in **Figure 1202.04.01**. The sample has been electropolished before anodising.

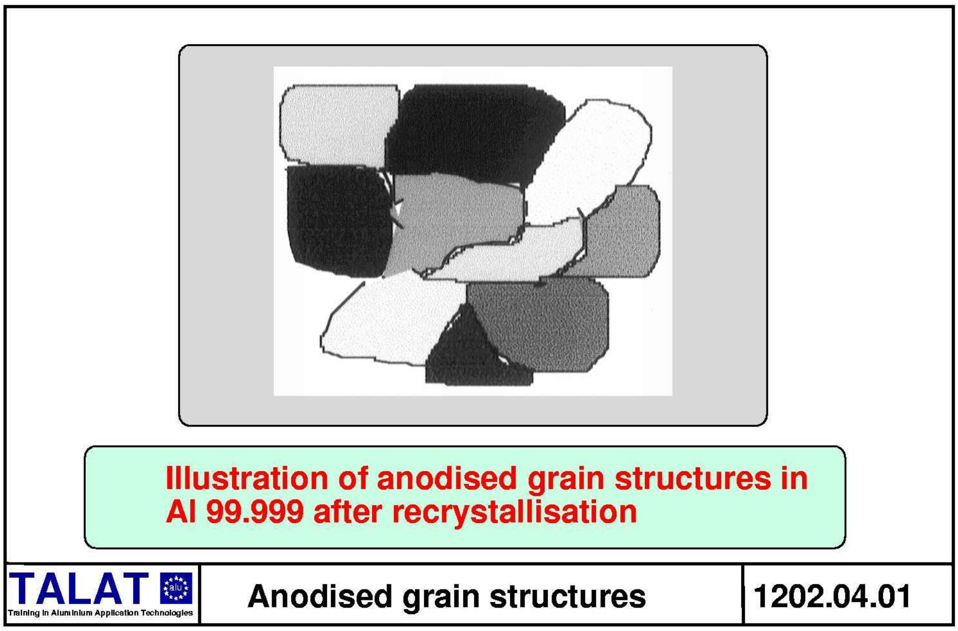


Illustration of anodised grain structures in Al 99.999 after recrystallisation

TALAT Training in Aluminium Application Technologies | **Anodised grain structures** | **1202.04.01**

The cold worked structure of aluminium can be better examined by TEM that reveals dislocation and grain boundary structure. But this is a more microscopic scale to follow the structure evolution. It depends on the degree of information the investigator needs.

1202.04.02 Wrought alloys

Conventionally produced aluminium alloys originate from a cast ingot from which all subsequent mechanical and thermal processing represent different degrees of change of the as-cast structure. Modifications are relatively minor for large wrought forms like forged pieces, thick plates and heavy extrusions that are hot worked. The modifications become greater as the total amount of reduction in original cross-sectional area is increased by hot and cold working and as the frequency of the thermal treatments is increased [13]. The most visible modifications may consist of :

- solution of more soluble phases or coalescence to reduce their surface energy
- precipitation of elements at elevated temperature that had supersaturated the as cast solution
- mechanical fragmentation of brittle intermetallic phases and their alignment along the principal direction of working
- process of recovery and recrystallisation as a consequence of deformation.

As it is reported above, wrought alloys are divided in eight major classes according to their alloying elements and thus each class has a different microstructure. In this chapter typical microstructural features are described for the major classes. Another division concerns the strengthening by work hardening or by precipitation that is non-heat treatable (1xxx, 3xxx, 4xxx, 5xxx) and heat treatable alloys respectively (2xxx, 6xxx, 7xxx).

1xxx - In this family it is very common to find phases of aluminium-iron or aluminium-iron-silicon because iron and silicon have a low solubility in aluminium and they are ever present as impurity elements. So it is easy to reveal phases like $FeAl_3$, Fe_3SiAl or $Fe_2Si_2Al_9$ by etching the sample with a solution of 0.5% HF. If the interest is addressed to the grain structure, also in this case the anodisation by the Barker's reagent is recommended.

In a number of alloy systems the most important microstructural changes involve the dislocation and subgrain structures. They depend of course from the degree of deformation and the temperature and can represent recovery, involving the rearrangement of dislocation into lower energy configuration or recrystallisation processes. In this system (1xxx), and in 3xxx and 5xxx families, the recrystallized condition is fundamental because they are supplied in the fully recrystallised state to be used in forming applications. So TEM investigation is necessary in these cases to understand the kinetic and the evolution of these processes.

3xxx - The addition of manganese to the chemical composition creates phases like $(Mn,Fe)Al_6$ or $(Mn,Fe)_3SiAl_{12}$ that can be revealed by a solution of 10% of phosphoric acid. The grain structure obtained by work hardening or by annealing are usually revealed by anodisation.

4xxx - Most of the alloys belonging to this family are used for welding and brazing filler materials where they are remelted. In any case, the good joining properties depend on the initial wrought structure. The as-cast phases are usually Si and $Fe_2Si_2Al_9$. During thermal treatment Si coalesces while the iron phase may remain unchanged. These particles are etched by a solution of 0.5% HF.

5xxx - Magnesium is largely soluble in aluminum compared to the other elements, but the content in excess can appear as eutectic Mg_2Al_3. After cold rolling and annealing they can be found at the grain boundaries or after cold working they can precipitate on deformation bands. In both cases the structure can be revealed by an etching of 10% H_3PO_4. At the same time as Cr is a frequent additive in this series, $Cr_2Mg_3Al_{18}$ may appear as a fine dispersoid.

6xxx - This family takes the main advantages from the strengthening due to the precipitation of Mg_2Si. So the etching will reveal the iron rich phases like Fe_3SiAl_{12}, $Fe_2Si_2Al_9$ that are insoluble and the coarse precipitates or the excess soluble precipitates (0.5%HF). The initial stage of precipitation is visible only at the TEM. The sequence of ageing is the following :
s.s.s (supersaturated solid solution)$\rightarrow$ GP$\rightarrow$ β' (Mg_2Si) $\rightarrow$ β (Mg_2Si), see **TALAT 1204** for more details.

Also the **2xxx** and **7xxx** systems are characterised by ageing sequences that can be extensively revealed only by TEM. In fact the precipitation process is strictly connected to defect density like vacancies and dislocations, and the several intermediate phases that are involved complicate the nature of observation. So only TEM can solve this scale of microstructure.

2xxx - This group is rather complex because of the many additives used for strength, corrosion resistance or grain control. So the system is usually multiphase especially in the as cast state. After homogenisation, it is probable to find Al_2CuMg and the iron containing phase Al_7Cu_2Fe, visible by etching with 10% H_3PO_4. The sequence of precipitation in this system is the s.s.s $\rightarrow$ GP$\rightarrow$ θ'' $\rightarrow$ $\theta'$$\rightarrow$ θ (Al_2Cu). As the copper content is usually 3.5-5% the last two phases of the sequence are visible at the optical microscope by etching with Keller's reagent.

7xxx - This class of aluminium alloys contains zinc, magnesium and copper as major elements as well as additives like Cr, Zr, Mn and Fe and Si. Thus the number of constituents and precipitation systems is further increased. These may be some of the constituents : Al_7Cu_2Fe, $Cr_2Mg_3Al_{18}$, $(Fe,Cr)_3SiAl_{12}$, Al_3Zr and the precipitation sequences follow the 2xxx and 6xxx series. The Keller's reagent may reveal most of them. Concerning the grain or subgrain structure, it can be revealed by a solution of 10% H_3PO_4, but of course a deeper investigation always need TEM [13].

1202.04.03 Foundry alloys

Aluminium alloy castings can be produced by virtually all casting processes in a range of compositions possessing a wide variety of useful engineering properties. For large production quantities, high-pressure die, permanent mould and sand are the most important casting processes. Smaller quantities of castings are produced in plaster, investment and composite material moulds. Commercial aluminium casting alloys include both heat treatable and non-heat treatable compositions. Castings made from non-heat treatable alloys are not appreciably affected by heat treatment and are usually marketed in the as-cast conditions except for the spheroidisation of Si in the Al-Si system to improve ductility. Alloys that respond to heat treatments provide improved combinations of mechanical properties [13].

2xx.x - usually this alloy system has a marginal castability relative to most of the alloy containing silicon, and also limited fluidity. In addition pressure tight parts of intricate design are difficult to obtain. The integrity of the part can be revealed by optical microscopy an as polished sample, without etching (see fig. 105 in ref. 4). The constituents are mainly the same as for the wrought system.

5xx.x - this family is characterised by excellent corrosion resistance, good machinability and attractive appearance when anodised. Also in this case more care in gating and greater chilling respect to Si containing alloys are required to produce sound castings. An etching by 0.5% HF may reveal the sand cast structure with the main constituent particles [see fig. 144-144 in ref. 4].

Al-Si (3xx.x and 4xx.x) - Alloys with Si as a major alloying element are the most important commercial casting alloys because of their superior casting characteristic in comparison to other alloys. They also have high corrosion resistance, good weldability and low specific gravity. The microstructural features consist mainly of network of silicon particles deriving from the interdendritic aluminium-silicon eutectic, etched by 0.5%HF. A solution treatment spheroidises the eutectic and ageing produces precipitates in the regions surrounded by Si [see fig. 109-141 in ref. 4].

7xx.x - Generally these alloys have good machinability and high melting points, making them suitable for castings that have to be assembled by brazing. They can be sand cast with attention to a good feeding ; hot cracking make them not suitable for permanent mould casting [13]. The suitable etching solution is the same as above.

8xx.x - The tin containing alloys were developed for bearings with high load-carrying capacity and fatigue strength. All the alloys can be cast in both sand and permanent mould.

1202.05 References

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1202.06 List of Figures

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