Metallographic preparation of aluminium and aluminium alloys

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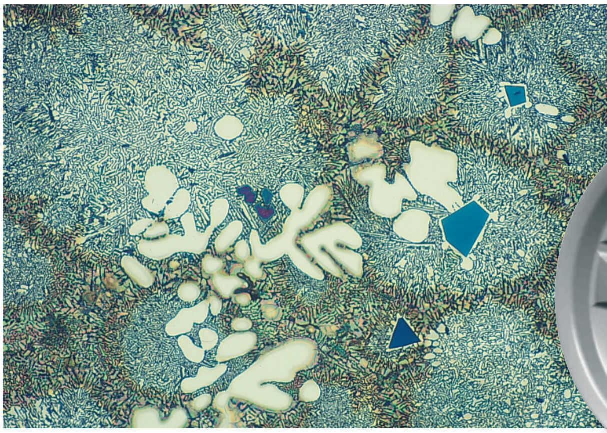
Application Notes

Unlike other long established metals such as iron and copper, aluminium is a relatively young metal and has been known only since the beginning of the 19th century. In 1886 a production method for aluminium was developed which is still used today: the Hall-Heroult-Process.

Aluminium has a silvery white appearance, is very light and is used as pure metal or alloyed for a multitude of different applications. Only very small amounts of alloying elements can increase the strength, and due to their low density, aluminium alloys are especially suited for applications in the aircraft and aerospace industry. Also aluminium alloys are used frequently in the automobile industry to promote weight reductions.

The high corrosion resistance of aluminium is based on a passive film of aluminium oxide that is intimately connected to the surface. This oxide film is capable of renewing itself spontaneously when the surface is damaged. Therefore aluminium is suited for polished and brushed surfaces and for anodising in various colours, which makes it an interesting material for the building industry.

Additional properties of aluminium are its high heat conductivity and its easy formability, either by casting, hot or cold working or machining. Aluminium is non-toxic and neutral regarding taste and is therefore the preferred material for the food and packaging industry, for instance for cans and foils or machines for bakeries and beverage pumps.



Aluminium-silicon cast, colour etched with molybdic acid, 200x.

New alloys such as Al-Li and Ti-Al are manufactured by powder metallurgical process and aluminium is also used as matrix metal for composites. Being a very multifaceted material, aluminium will in future open up more fields for new applications.

Metallography of aluminium is used in quality control for grain size determination and for micro structure defects on the polished and etched specimen. In addition, the sample is checked for impurities such as oxides or zirconium aluminides (Fig. 2). Cast material is evaluated for shape and distribution of phases and possible porosity. In wrought material, defects from the rolling and extrusion process are investigated and plating thicknesses measured.

Difficulties during metallographic preparation

- Pure aluminium is very soft and prone to mechanical deformation and scratching.
- Silicon carbide and diamond particles can be pressed into the specimen surface (Fig.1)
- Severely worked and deformed wrought alloys are difficult to contrast

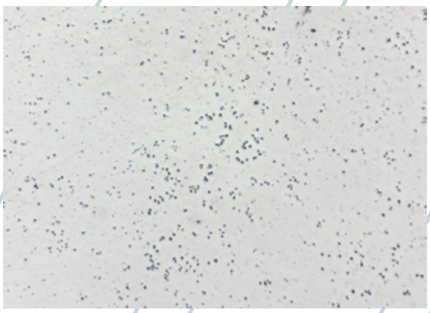


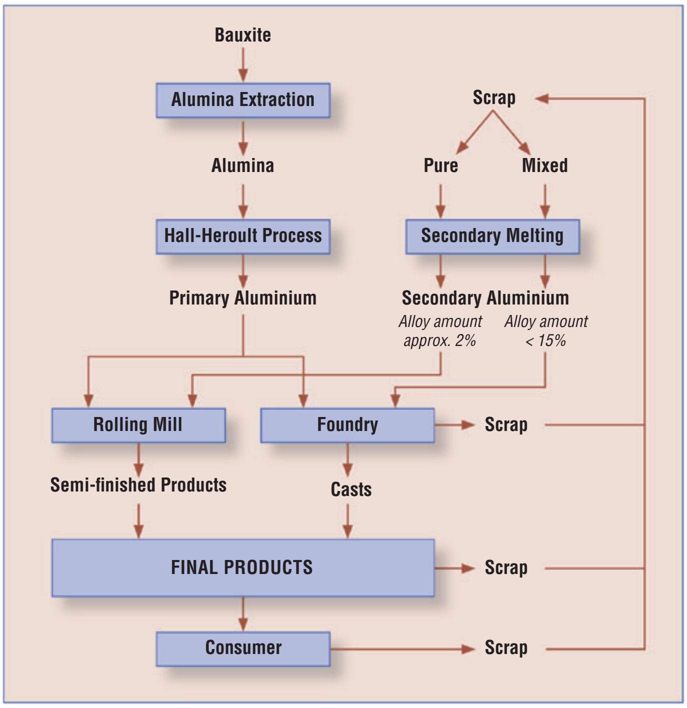
Fig.1: Embedded diamond particles in pure aluminium after polishing with 3 µm, 200x.

Fig. 2: Oxide in the surface of an aluminium pressure die casting, 50x.

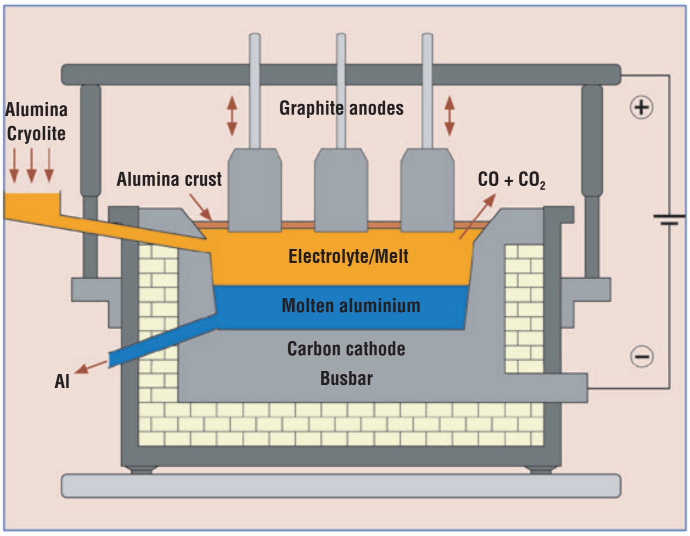
Solution

- Plane grinding with the finest possible SiC-Foil/Paper
- Diamond polishing and/or final polishing need to be long enough to remove all embedded particles
- Final polishing with Collodial Silica suspension
- Anodising with Barker's reagent

Production and application of aluminium



Simplified diagram of the aluminium production process.



Schematic drawing of the electrolysis producing pure aluminium.

Aluminium is one of the most frequently occuring metals in the earth's crust. It is not found in its pure state but combined in the form of chemical compounds. An economical extraction is only possible from bauxite which consists of 60% aluminium oxide as hydroxide (Al_2O_3 + H_2O), the rest are metal oxides (Fe_2O_3, SiO_2, TiO_2). The production process is complex and energy intensive and is mainly based on two steps that are briefly described in the following.

Extraction of pure alumina (aluminium oxide, Al_2O_3) from bauxite

Alumina recovery begins by crushing and finely grinding the bauxite and heating it with sodium hydroxide under pressure. In this process a water soluble sodium aluminate is formed together with undissolved residues of iron, titanium and silicon, called "red mud" that is separated by sedimentation. To the highly diluted sodium aluminate solution "seed crystals" of fresh aluminium hydroxide are added to initiate the precipitation of pure aluminium hydroxide ($Al(OH)_3$). Through calcination at 1200°C the water is removed from aluminium hydroxide and pure anhydrous alumina (aluminium oxide) remains.

The Hall-Heroult process: Converting alumina to aluminium

The reaction chemistry of pure alumina requires an electro-chemical process to extract aluminium from its oxide. As the melting point of aluminium oxide is very high, 2050°C, it is mixed with cryolite which reduces the melting point to 950°C. In addition, the cryolite increases the conductivity and consequently the electricity yield.

The electrolysis takes place in a large carbon or graphite lined steel container that contains steel rods for conducting electricity. The carbon clad walls and floor form the cathode and the aluminium oxide-cryolite melt is the electrolyte. Carbon blocks hang on a rack over the melt function as anodes. During the electrolysis the carbon of the anode reacts with the oxygen of the alumina and in a secondary reaction, metallic aluminium is produced with the formation of carbon dioxide:

$$2\,Al_2O_3 + 3\,C \rightarrow 4\,Al + 3\,CO_2$$

The molten aluminium has a higher density than the electrolyte-melt and settles at the bottom of the pot. It is tapped once a day and cast into pigs. With this process, aluminium of 99.0 – 99.9% purity is produced of which the largest part is used for the production of aluminium alloys.

Through an additional electrolysis the purity of the aluminium can be increased to 99.98% for semi-finished products and to 99.99% for pig casts. This pure aluminium has a very high corrosion resistance and is particularly well suited for anodising. It is used for brightening products such as decorative trim, reflectors, foils for electrolyte-capacitors and for tubes in the chemical and food industry. As the cost of aluminium increases with increasing purity, the purity of a specific aluminium used for a product is determined by the requirements of its application.

The diagram shows the cycle of aluminium with its production steps. The main products are wrought alloys for semi-finished products containing up to 2% alloying elements and cast alloys with up to 15% alloying elements.

There are numerous **aluminium alloys** and they are categorised as wrought and cast alloys. These two groups are differentiated again by heat-treatable and non-heat-treatable alloys. The addition of very small amounts of alloying elements produce an increase in tensile strength, yield strength and hardness when compared with the mechanical properties of pure aluminium. The most important alloying elements are Si, Mg, Cu, Zn and Mn. Combinations of alloying elements with aluminium or with each other (Mg_2Si, Al_2Cu, $MgZn_2$ and Al-Fe-Si compounds) influence the workability. These mostly eutectic compounds have to be finely dispersed first through a hot working process before the alloy can be cold worked.

Lamps with reflectors of highly polished aluminium.

The most important process to improve the mechanical properties of aluminium alloys is age hardening. It requires a solid solution with a decreasing solubility of the alloying elements with falling temperatures, for instance AlCuMg and AlMgSi.

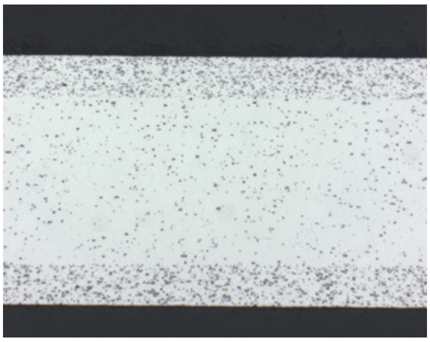
Natural age hardening (example AlCuMg). After solution annealing the workpiece is quenched and consequently the precipitation of the Al_2Cu in the solid solution is suppressed. The workpiece is then left to age in ambient temperature. During this process the aluminium lattice precipitates the copper from the supersaturated solution. The resultant strain produced in the aluminium lattice leads to an increase in strength and hardness. Natural hardening takes approx. 5-8 days.

In the **artificial age hardening** process the dynamics are the same as described above. However, ageing takes place at an elevated temperature. For instance, for an AlMgSi alloy, ageing occurs in 4-48 hrs at 120-175°C after solution annealing and quenching. The precipitation of the Mg_2Si phase produces internal strain in the aluminium lattice which results in an increase of strength and hardness.

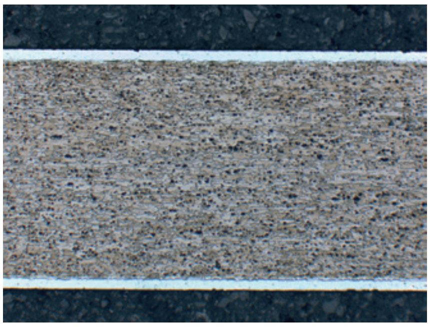
Wrought alloys

Billets for rolling and ingots for pressing are produced in a continuous casting process. In many cases casting is followed by annealing for homogenisation. Transformation of the cast structure into a wrought structure is effected by hot rolling, extrusion or forging. Only after this hot forming, in which the embrittling eutectic grain and cell boundary structure is broken up, is the cold working of wrought alloys possible (Fig. 3-5).
The main alloying elements for wrought aluminium alloys are copper, magnesium, zink and manganese. Silicon and iron affect the mechanical properties and corrosion resistance and can either be impurities or alloying elements, depending on the requested purity and application.

Wrought alloys are for example used for plates in mechanical engineering and mould construction, for rolled products such as sheet and strip and for plated products like radiators and heat exchangers. Plated sheets which have to combine specific mechanical properties and high corrosion resistance have a core billet of an aluminium alloy that is covered on both sides with pure aluminium and rolled. These plated sheets are used for specific semi-finished products for aircraft construction or for decorative applications such as trim and reflectors.



Plated sheet, core: aluminium alloy 3503, plating: aluminium alloy 4104, unetched 100x.



Plated sheet, core: aluminium alloy 2024, plating: aluminium 99.5%, etched with Dix and Keller, 50x.

High strength aluminium wrought alloys are used in mechanical engineering, conveying and electro technical applications and for sports and leisure products such as bindings for snowboards, ski poles and gears for mountain bikes etc.
Aluminium is increasingly used as matrix material for composites, especially for fibre reinforced aluminium in the aircraft and aerospace industries.

Non hardening alloys are work hardened through cold working		
Main alloying element	*Serial number*	*Examples of alloys*
More than 99% pure, with traces of Cu, Fe, Si	1xxx	Al 99,0; Al 99,5; Al 99,85
Mn	3xxx	Al-Mn; Al-Cu-Mn-Mg
Mg	5xxx	AlMg3; Al-Mg-Mn-Cr
Other elements	8xxx	Al-Fe-Si; Al-Li-Mg-Cu
Hardening alloys are hardened through natural and artificial hardening		
Cu	2xxx	Al-Cu-Si-Mg
Si	4xxx	Al-Si-Mg
Mg, Si	6xxx	Al-Mg-Si
Zn	7xxx	Al-Zn-Mg-Cu

Fig. 3: Aluminium alloy 2024, cast, showing eutectic precipitation on grain boundaries, unetched, 200x.

Fig. 4: As Fig. 3, homogenised, unetched, 200x.

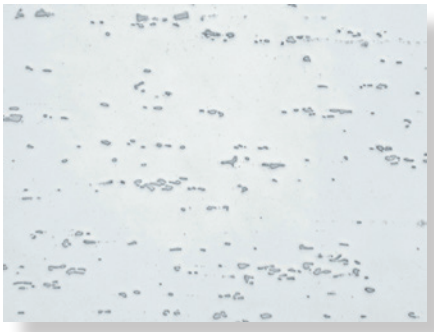


Fig. 5: As Fig. 4, hot rolled, unetched, 200x.

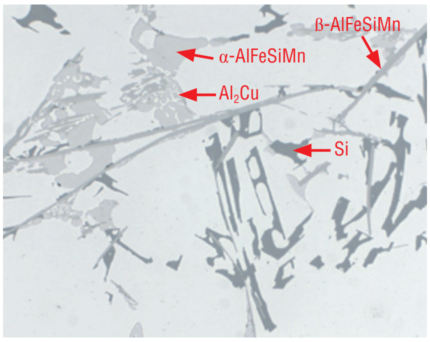


Fig. 6: AlSi6Cu4 cast, the various phases can be identified by there typical colour.

Cast alloys
Aluminium casts are mainly alloyed to improve the mechanical properties and are differentiated according to their main alloying elements silicon, magnesium and copper. Alloy contents that exceed the saturation of the solid solution are precipitated as pure metal such as silicon or as eutectics and inter metallic phases.

Silicon increases the castability of aluminium, and in eutectic alloys, such as AlSi12, small amounts of sodium are added before casting to refine the eutectic. In this refining process, instead of precipitating as coarse needles or plates (Fig. 7), silicon forms a very fine eutectic with the α-solid solution (Fig. 8). The effect of hardening in these alloys is very low and therefore magnesium is added so that they can be age hardened.

Cast alloys are produced by sand- chill- or die casting. Alloys with specific properties are used in various product groups for the fabrication of pistons, slide bearings, parts for mechanical engineering, cylinder heads, brake shoes etc.

Some of the more important cast alloys and their properties are listed:

AlSi10Mg	Age hardening, vibration and corrosion resistant
AlSi5Cu1	Age hardening, good castebility, for welding, for thin section casting
AlMg3	Resistant against sea water
AlSi25+ Cu Ni	Age hardening, special alloy, for pistons, wear resistant due to high Si content
AlMgSiPb	Suitable for machining
AlSi9Cu3	Well castable universal alloy, most important alloy for pressure die casting

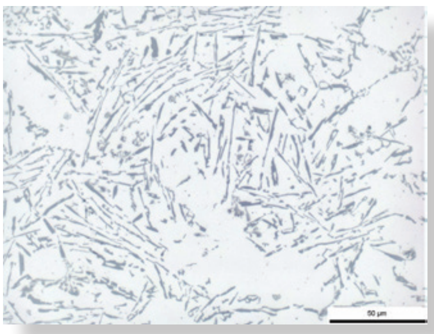


Fig. 7: Aluminium-silicon cast, unrefined, 500x.

Difficulties during the preparation of aluminium and aluminium alloys

With increasing purity, aluminium becomes softer and more susceptible to mechanical deformation and scratches. Consequently, grinding can cause already deep deformation in high purity aluminium.
Grinding and polishing abrasives can be pressed into the surface. With increasing alloying content aluminium becomes harder and cast alloys are relatively easy to prepare. It needs to be emphasised though, that the aluminium matrix has to be well polished in order to avoid errors in the structure interpretation (Fig. 9 and 10).

Fig. 9: Aluminium-silicon cast, after polishing with 3µm diamond small scratches are still visible, 200x.

Fig.10: Structure as in Fig. 9, but fine polished with OP-U suspension. The matrix is well polished and the eutectic has more contrast, 200x.

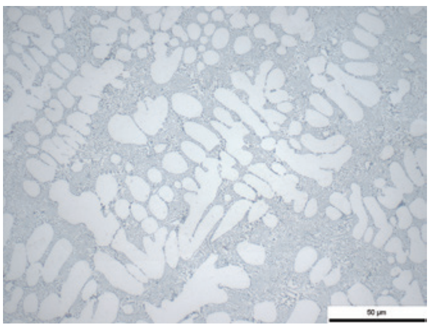


Fig. 8: Aluminium-silicon cast, refined, 500x.

Recommendations for the preparation of aluminium and aluminium alloys

For **sectioning** of aluminium a hard silicon carbide cut-off wheel is used that is generally suitable for non-ferrous metals. For **mounting** resin a phenolic resin is in most cases sufficient. Thin sheets and foils and specimens for anodising are best mounted in slow curing epoxy resins, whereby the samples should protrude from the back of the mount to serve as electrical contacts.

Mechanical grinding and polishing
It is recommended that plane grinding is carried out with the finest possible grit in order to avoid any excessive mechanical deformation. Hardness, size and number of samples have to be taken into account, but even with larger samples of pure aluminium, 500# is sufficient for plane grinding. Large cast parts of aluminium alloys can be ground with 220# or 320#. It is important that the force for grinding is also very low to avoid deep deformation and reduce friction between grinding foil/paper and sample surface; this can be relatively high for pure aluminium.

Diamond polishing has to be carried out until all deep scratches from grinding have been removed.
In case water soluble constituents have to be identified, polishing with water free diamond suspension and lubricant is recommended.
If diamond particles have been pressed into the surface during polishing the following final polish with silicon dioxide suspension can be relatively long (See Fig.1). Very often one can see already with the naked eye bright and dull areas on the samples surface. This is an indication that the final polish is not yet sufficient. It needs to be pointed out that the embedded particles can lead to erroneous interpretations of the structure.

Section of a mechanical engineering part with aluminium cast components.

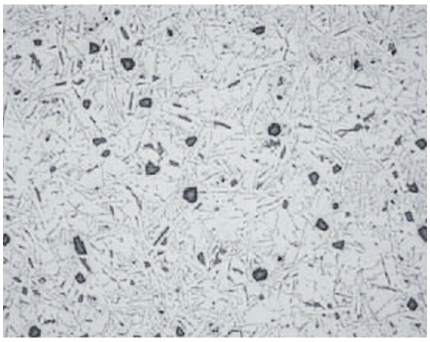


Fig.11: Aluminium-silicon cast polished too long with OP-S suspension, silicon precipitates stand in relief, 100x.

After 1 minute polishing with OP-U suspension the result is checked in the microscope. If necessary the polish should be continued for another minute and the result checked again. It is recommended to continue this polish /check sequence until the required quality of the result has been achieved.

(Approx. 20-30 sec before the end of polishing, water is poured onto the polishing cloth to rinse the sample as well as the cloth. Then the sample is washed again with clean water from the tap and then dried. *Note:* Polishing too long with silicon dioxide suspension OP-S NonDry can cause a pronounced relief, see Fig.11).

The following data are for the automatic preparation of mounted specimens, 30 mm.

Aluminium - Silicon Cast

Grinding

Step		PG	FG
Surface		MD-Molto	MD-Largo
Abrasive	Type	Diamond	Diamond
	Size	#220	9 µm
Suspension / Lubricant		Water	DiaPro Allegro/ Largo 9
Rpm		300	150
Force (N)/ specimen		25	30
Time (min)		Until plane	4

Polishing

Step		DP	OP
Surface		MD-Mol*	MD-Chem
Abrasive	Type	Diamond	Collodial Silica
	Size	3 µm	0.04 µm
Suspension / Lubricant		DiaPro Mol R3	OP-U NonDry
Rpm		150	150
Force (N)/ specimen		25	15
Time (min)		3	1

**Alternatively MD-Dac.*

Electrolytic Polishing is especially suited for pure aluminium and wrought alloys. It leaves a scratch free surface and is often used in quality control as it delivers fast and reproducible results. For routine grain size determination at 100x, pre-grinding with 1000# SiC-Foil/Paper is sufficient. Rolled or drawn surfaces do not need any grinding or polishing. For pure aluminium and precise examinations of grain shapes the samples have to be fine ground to 2000# and sometimes even to 4000# before electrolytic polishing. Due to the many different phases in cast alloys, they are not suited for electrolytic polishing.

Pure Aluminium

Grinding

Step		PG	FG 1	FG 2	FG 3
Surface		Foil/Paper*	Foil/Paper*	Foil/Paper*	Foil/Paper*
Abrasive	Type	SiC	SiC	SiC	SiC
	Size	#320/500	#800	#1200	#4000
Suspension / Lubricant		Water	Water	Water	Water
Rpm		300	300	300	300
Force (N)/ specimen		25	25	25	25
Time (min)		Until plane	0.5	0.5	0.5

Polishing

Step		DP	OP
Surface		MD-Mol**	MD-Chem
Abrasive	Type	Diamond	Collodial Silica
	Size	3 µm	0.04 µm
Suspension / Lubricant		DiaPro Mol R3	OP-U or OP-S
Rpm		150	150
Force (N)/ specimen		25	15
Time (min)		4	2-5

** To avoid coarse scratches, the SiC-Foil/ Paper can be rubbed with wax before grinding.*

***Alternatively MD-Dac*

Pure Aluminium, alternative method

Grinding

Step		PG	FG
Surface		Foil/Paper*	MD-Pan
Abrasive	Type	SiC	Diamond
	Size	#320	15 µm
Suspension / Lubricant		Water	DiaPro Pan 15
Rpm		300	150
Force (N) / specimen		25	25
Time (min)		Until plane	5

Polishing

Step		DP	OP
Surface		MD-Dac 3 or MD/Dur 3	MD-Chem
Abrasive	Type	Diamond	Collodial Silica
	Size	3 µm	0.04 µm
Suspension / Lubricant		DiaPro Dac 3/Dur 3	OP-U or OP-S
Rpm		150	150
Force (N) / specimen		25	15
Time (min)		4	2-5

**To avoid coarse scratches, the SiC-Foil/Paper can be rubbed with wax before grinding.*

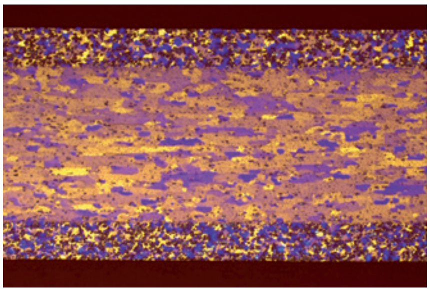


Fig.12: Plated sheet, anodised, grain areas are clearly visible and suited for automatic image analysis, polarised light with λ¼-plate, 100x.

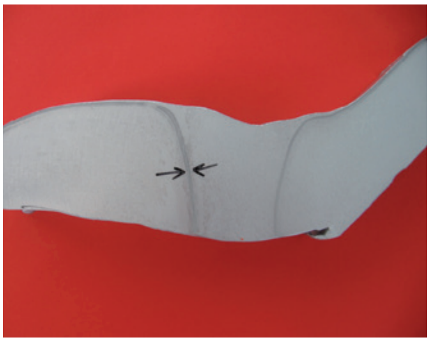


Fig.13: Pressed part, macro etching, primary and heterogeneous precipitates are revealed that have a negative effect on the surface of the final product.

Electrolyte:	A2
Mask:	2 cm²
Voltage:	39 V
Flow rate:	10
Time:	20 sec.

After the polish, anodising with Barker's reagent gives a colour contrast that is particularly suited for grain size evaluation. To obtain the colour effect, polarised light with a λ¼ sensitive tint plate is used (Fig.12).

Electrolyte:	200 ml water 10 ml fluoroboric acid (35%) (Barker's)
Mask:	2 cm²
Voltage:	24 V
Flow rate:	8
Time:	1-2 min.

Etching and structure

Macro etchants are used for grain size evaluation; also to show flow lines from extrusion (Fig.13) and for revealing weld seams. Before etching, the sample has to be ground to 1200# or 2400#.

The many alloying possibilities of aluminium result in a large variety of different phases that cannot always be clearly identified in some of the multi component alloys. Some of the well known phases have the following characteristic colours (see also Fig.6):

Si	Grey
Mg_2Si	Tarnished dark blue during polishing (in cast: Chinese script)
Al_2Cu	Pinkish-brown, copper coloured
Al_6Mn	Light grey

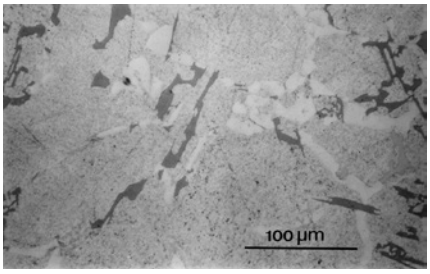


Fig.15: Experimental aluminium alloy with 6% Si and 10% copper, unetched.

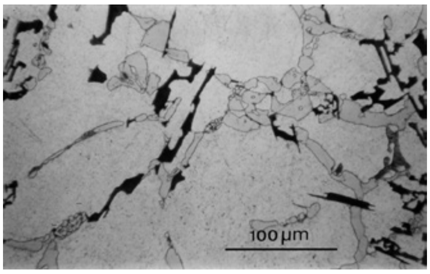


Fig.16: Same as Fig.15 but etched for 30 sec with 1g molybdic acid in 200 ml water + 6 g ammonium chloride. The silicon is dark blue and is distinguishable from the greyish $CuAl_2$.

Depending on the alloying content, these eutectic phases can sometimes be recognised by the typical shape of their eutectic. The relevant literature lists etchants which can be used to identify these phases (Fig.15 and 16).

Wrought alloys with a heavily deformed structure such as foils or very thin sheets are difficult to contrast with chemical etchants; this can make the automatic image analysis difficult. It is recommended to anodize the sample with Barker's reagent and then evaluate the structure "manually" with the microscope as sometimes, even with Barker's reagent, the contrast is not sufficient for an automatic image analysis. It should be pointed out that certain specifications for wrought alloys require "manual" grain size determination, as the image analysis is not precise enough.

Etching solutions

Note: When working with chemicals the standard safety precautions have to be observed.

Macro etching

For pure aluminium	90 ml water 15 ml hydrochloric acid 10 ml hydrofluoric acid
Deep etching to reveal the primary dendritic structure	100 ml water 10-25 g sodium hydroxide

Micro etching

Flick's reagent: Grain boundary etching for most types of aluminium and alloys	90-100 ml water 0.1-10 ml hydrofluoric acid
Dix and Keller reagent: Grain area etching for Al-alloys with copper, also suitable for pure aluminium.	190 ml water 5 ml nitric acid 10 ml hydrochloric acid 2 ml hydrofluoric acid
Colour etchants:	Molybdic acid solution according Klemm or Weck

Summary

Its low density, high strength and corrosion resistance make aluminium and its alloys the material of choice for many applications in, among others, the automobile, aircraft/aerospace and packaging industry. Metallography is used in quality control for grain size determination, evaluation of phases, impurities and mechanical defects. Pure aluminium is very susceptible to deformation and therefore grinding should not be carried out with coarse grits. A very thorough final polishing with silicon dioxide suspension is necessary to ensure that embedded diamond particles are completely removed from the sample surface. Aluminium cast alloys are polished relatively easily. For grain size evaluation anodising with Barker's reagent is particularly suited as it results in a better contrast than chemical etching. Different phases in cast alloys can either be identified by their characteristic colour or by etching with specific solutions that attack certain phases preferentially.

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Fig.17: Burr on a solder spot of a heat exchanger, pre-etched with sodium hydroxide, colour etched with potassium permanganate according to Weck.

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