

The Eloxal process

Time required: 40 min

Aims of the experiment

- To protect aluminium as a light and robust material.
- To understand the process of protecting aluminium.
- To apply an oxide layer onto aluminium.
- To understand the Eloxal process as a large-scale industrial process.

Principles

In the corrosion of metals, electrochemical processes play a significant role. For example, pure iron rusts in connection with water and oxygen in the air. The mechanical and chemical properties of the material change. The material is brittle and full of cracks.

In order to protect metals, corrosion protection has been developed. One possibility is to coat the metal with oils, greases or paints, which excludes water and oxygen from penetrating inward.

Another protection mechanism is to coat or short-circuit the metal with a less precious metal. This metal is then called the sacrificial anode since it is more reactive to external influences and is the first to dissolve.

Aluminium and zinc can protect themselves against the effects of weather. They form adherent oxide layers on their surfaces.

For aluminium materials that are under a particularly serious load, the so-called Eloxal process (**E**lectrolytic **O**xidation of **A**luminium) was developed. In this process, the natural oxide layer is strengthened. Anodised or eloxed aluminium has an elevated degree of protection against mechanical influences and is weather-proof and corrosion-resistant. The oxide coating thickness can then be applied up to a few micrometres in thickness, depending on the need. For example, treated aluminium is frequently used as a robust material used in space technology.

But what happens in the process of anodisation?

First of all, the degreased surface is rendered anodisable by etching. Now, during the anode reaction aluminium becomes oxidised to aluminium(III), whereas at the cathode hydrogen and water are released from the acid. At the anode, the released aluminium(III) reacts further with the water to form aluminium oxide, which deposits at the anode and constitutes the actual process of anodisation. The process is concluded by the sealing of the pores with boiling water or water vapour. The half reactions are shown below.

			Redox potential
Anode	$2 \text{Al}^0 \rightarrow 2 \text{Al}^{+III} + 6 \text{e}^-$		-1.66 V
	$2 \text{Al}^{+III} + 9 \text{H}_2\text{O} \rightarrow \text{Al}_2\text{O}_3 + 6 \text{H}_3\text{O}^+$		
Cathode	$6 \text{H}_3\text{O}^{+II} + 6 \text{e}^- \rightarrow 6 \text{H}_2\text{O} + 3 \text{H}_2^0$		0 V
Total	$2 \text{Al} + 3 \text{H}_2\text{O} \rightarrow \text{Al}_2\text{O}_3 + 3 \text{H}_2$		1.66 V

In this experiment, anodised aluminium is prepared. Then the properties of natural aluminium and anodised aluminium are compared. Here, mechanical and physical properties will be investigated.

Risk assessment

CAUTION! Sodium hydroxide solution and sulfuric acid are corrosive and cause eye damage. Wear protective goggles and a laboratory coat.

Acetone is highly flammable. Keep all sources of ignition away. To be safe, use only in the fume hood.



Fig. 1: Set-up and materials for the experiment.

Sulfuric acid, dilute, approx. 2 N	
	<p>Hazard warnings</p> <p>H290 May be corrosive to metals. H315 Causes skin irritation. H319 Causes serious eye irritation.</p> <p>Safety information</p> <p>P280 Wear protective gloves / eye protection.</p> <p>P305+P351+P338 IF IN EYES: Rinse carefully with water for several minutes. Remove contact lenses if present and if possible to do so. Continue rinsing.</p> <p>P337+P313 If eye irritation persists: Get medical advice/attention.</p> <p>P302+P352 IF ON SKIN: Wash with plenty of water and soap.</p>
<p>Signal word: Hazard</p>	

Sodium hydroxide, 1 mol/l	
	<p>Hazard warnings</p> <p>H314 Causes severe skin burns and eye damage. H290 May be corrosive to metals.</p> <p>Safety information</p> <p>P280 Wear protective gloves/protective clothing/eye protection.</p> <p>P303+P361+P353 IF CONTACT WITH SKIN OCCURS (or hair): Remove/take off all contaminated, soaked clothing immediately. Rinse skin with water/shower.</p> <p>P305+P351+P338 IF IN EYES: Rinse carefully with water for several minutes. Remove contact lenses if present and if possible to do so. Continue rinsing.</p> <p>P310 Call a POISON CENTER or doctor/physician immediately.</p> <p>P301 + P330 + P331 IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.</p> <p>P406 Keep in corrosion-resistant containers with corrosion-resistant linings.</p>
<p>Signal word: Hazard</p>	

Acetone	
	<p>Hazard warnings</p> <p>H225 Highly flammable liquid and vapour. H319 Causes serious eye irritation. H336 May cause drowsiness or dizziness.</p> <p>EUH066 Repeated contact can lead to brittle or cracked skin.</p>
	<p>Safety information</p> <p>P210 Keep away from heat/sparks/open flames/hot surfaces. No smoking.</p> <p>P233 Keep container tightly closed.</p> <p>P305+P351+P338 IF IN EYES: Rinse carefully with water for several minutes. Remove contact lenses if present and if possible to do so. Continue rinsing.</p>
<p>Signal word: Hazard</p>	

Equipment and chemicals	
1	Electrochemistry demonstration unit, CPS.... 664 4071
1	Panel frame C50, two-level, for CPS..... 666 425
1	Electrochemistry table, CPS..... 666 472
1	Electrochemistry accessories set 664 401
1	Electrolysis cell.....from 664 401
1	Nail.....from 664 401
1	Grindstone.....from 664 401
1	Drip pan.....from 664 401
2	Connecting leads.....from 664 401
2	Crocodile clips.....from 664 401
1	Tweezers.....from 664 401
1	Carbon electrode.....from 664 401
1	Aluminium electrode.....from 664 401
1	Crucible tongs 667 035
1	Pasteur pipette, glass..... 665 9531
1	Rubber bulbs..... 665 9541
1	Beaker, 250 ml 664 130
1	Petri dish, 120 mm 602 740
1	Magnetic stirrer with hotplate 666 839
1	Stirring magnet..... 666 851
1	Acetone, 250 ml 670 0400
1	Sulfuric acid, approx. 2 N, 500 ml..... 674 7920
1	Sodium hydroxide solution, approx. 2M 673 8400
1	Water, pure, 1L..... 675 3400
Additionally:	
Eosin, fluorescein or other dye (not indigo!),	
Paper towels	

Set-up and preparation of the experiment

Set-up of the experiment

Suspend the demonstration unit (Fig. 3) and the electrochemistry table in the panel frame. Place the drip tray in the centre on the electrochemistry table. Fix both half cells of the electrolysis cell with the screws. The electrolysis cell is now sealed.

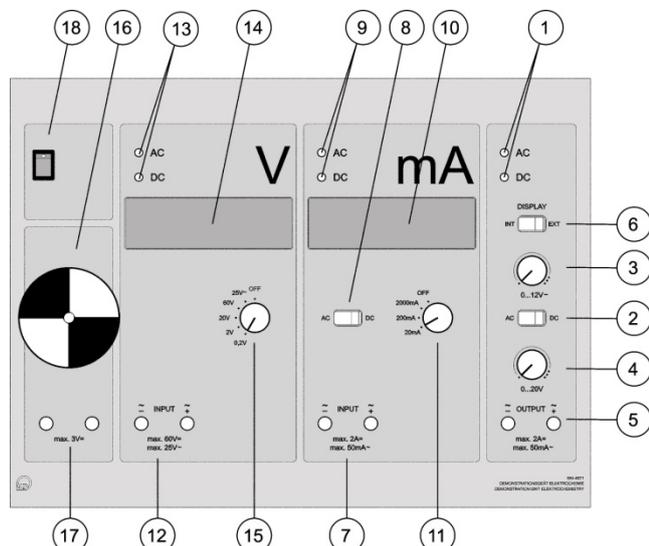


Fig. 2: Sketch of the demonstration unit.

Preparation of the Al electrode

Heat approximately 200 ml of distilled water to boiling in a beaker (250 ml) with a stirring magnet using the hot plate.

First, clean the aluminium electrode to be anodised with a grindstone. Then, transfer the electrode to a petri dish. Here, it will become coated with acetone for degreasing. Turn the electrode multiple times with the tweezers and after 1 minute place it on a paper towel to dry. From now on, the electrode may only be handled using the tweezers. The excess acetone can be left to evaporate in the fume hood.

For better adhesion of the oxide layer, the electrode can be etched beforehand with sodium hydroxide solution. To do so, the de-greased electrode is placed in the petri dish and coated with 1 molar sodium hydroxide solution. After a minute, turn the electrode.

Note: Significant hydrogen generation will occur.

Setting up the anodisation process

Place a carbon electrode and the prepared aluminium electrode into the third outermost groove using the tweezers. Place the electrolysis cell prepared as described in the centre of the electrochemistry table.

Connect the electrodes using a connecting lead and gripping clamp to the power supply (5) of the demonstration unit. Connect the carbon electrode to the negative output jack and the aluminium electrode to the positive output jack. Set the selector switch (6) to internal power source. Set the changeover switches (2) and (8) to direct current (DC). Set the rotating control (15) to the 20 V range. Set the rotary control (11) to 200 mA (see Fig. 2).

Performing the experiment

Anodisation

Turn on the demonstration unit using the switch (18). Sulfuric acid is the electrolyte. Fill it up to the 4 cm mark of the electrolysis cell. Raise the electrodes a bit in the process.

Set a direct current voltage of 12 V at the demonstration unit using rotary control (4). After about 10 min, the electrolysis can be ended by turning off the demonstration unit. Remove the aluminium electrode and rinse it with distilled water and inspect it for any obvious changes.

Sealing the anodised aluminium electrode

To seal the Al electrode, it is then dipped into boiling water using crucible tongs for 10 minutes (about 4 cm deep). After it cools down, inspect the electrode visually for any exterior changes.

To tint the work piece, a water-soluble dye can be added to the boiling water in advance. Then, proceed in the same way.

Examination of the hardness of the aluminium electrode.

To examine the hardness, the surface of the electrode is scratched with a nail with pressure. Scratching is done from the treated to the untreated position. What happens?

Examination of electrical conductivity

The flowing current is a measure of electrical conductivity. The higher the flowing current, the higher is the conductivity of the aluminium. To investigate the electrical conductivity of the oxide layer on the aluminium electrode, a very low voltage of no more than 0.6 V is set at the power supply (5).

Note: Short-circuiting can occur at higher voltages!

The untreated part of the electrode is connected to an output jack (5) using a gripping clamp. With a second connection lead connected to the power supply (5), the untreated side is now touched with the plug. What do you see on the amperemeter? Now, touch the treated side with the plug and observe the measurement display.

Investigation of corrosion properties

To examine the change in corrosion properties, two pipettes full of 1 molar sodium hydroxide solution are mixed with 15 pipettes full of distilled water in a petri dish. Now, place the electrode in it and observe.

Observation

Anodisation

During anodisation, the current initially drops, but increases over time again. At the carbon electrode, it can be observed that gas is being generated weakly. The surface of the aluminium electrode seems to be rough.

Sealing

When sealing, the non-anodised part is grey and matt. The anodised part seems unchanged.

Hardness

The nail penetrates the untreated side deeper into the aluminium surface than on the treated side.

Electrical conductivity

In the untreated part, current flows (see Table 1), and in the untreated part there is no current.

Tab. 1: Experimentally determined current measurements.

Treatment	Current measurement in a short circuit
Untreated	1.2 mA
Treated	0.018 mA

Corrosion properties

When the electrode is placed in dilute sodium hydroxide solution, gas generation is only seen at the untreated point. The treated point exhibits a slight generation of gas at the point damaged by the nail and at the corners and edges.

Results

The anodisation of aluminium changes the properties of the metal. First of all, the surface, because of the thicker oxide

layer, is much more resistant to external influences. The scratch test shows that the untreated side is much more damaged. The treated side has less damage.

The anodised surface does not conduct electric current. Thus it acts as an insulator. Furthermore, anodised aluminium is corrosion-resistant, which is different to untreated aluminium, as the corrosion test shows. Electrochemically-oxidised aluminium is therefore being produced in commercial amounts. In the process, the oxide layer thickness can vary and the material can be permanently tinted by adding water-soluble dyes, since the dye molecules are pulled into the pores during sealing.

Cleaning and disposal

Excess acetone should be left to evaporate in the fume hood or disposed of in the organic solvent waste canister B. All aqueous solutions can be disposed of in the laboratory drain with plenty of water.