WORKSHOP HOW-TO

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10. ANODISING ALUMINIUM

The chemistry of aluminium (and it's alloys) is interesting. Given that it's a reactive metal (more so than iron) one would expect it to naturally corrode more rapidly than it actually does. The reason it doesn't is that the initial formation of an oxide layer on the surface protects the base metal underneath. Aluminium oxide is a tough material (it's crystalised form is used as an abrasive - Allox), but normally it's formation in air results in an amorphous layer with little mechanical strength, it's a white powder that can easily be scraped off leading to further oxidation. Anodising on the other hand, is a process which forms a particularly structured and dense oxide layer which resists abrasion and thus protects the underlying metal. This layer is colourless, but it's possible to introduce a dye at one stage in the process to permanently colour the surface. Not all aluminium alloys can be easily anodised, cast aluminium (with a high silica content) is particularly difficult. The alloys I routinely use are off-cuts of unknown origin (well, they're cheap!) - some are free-cutting, some are not. However, I can say that I've had no difficulty in achieving an acceptable finish on all of those I've used so far.

Having made several parts in aluminium (alloy) for a telescope, I then had the problem of producing a durable black finish, preferably matt. Anyone who has tried to paint aluminium will know that it's extremely difficult to get the paint to adhere to the metal, even when using etching primers. The paint will easily chip leaving bright metal showing through. It was also important in my case that dimensions were maintained for close fitted parts - something clearly not possible with a coat of paint.

Anodising is an electro-chemical process which physically alters the surface of the metal to produce a tough oxide layer on the surface. During the anodising process the oxide layer is at first porous, and at this point it's possible to introduce a coloured dye. 'Porous' in this case refers to the molecular level rather than gaping holes - the dye thus needs to be soluble to penetrate this layer. The porous layer is then 'sealed' by boiling the part in water (which converts the oxide to a different crystalline chemical form) permanently trapping the dye beneath the surface. It should be noted that as the anodised surface is a layer of aluminium oxide, rather than metal, it exhibits a very high resistance to electric current (so don't try anodizing electrical contacts!)

METHOD:

There are several methods of producing the anodised finish, all differ a little in the chemistry used to achieve the porous/non-porous oxide layer. All use noxious chemicals (it's just that some are more noxious than others!). The method listed below involves the use of some fairly powerful reagents so in all cases wear eye-protection (goggles etc.,) and rubber gloves. There is no danger provided you are careful and handle the chemicals with respect - keep the containers covered and make sure youngsters can't accidentally knock them over. There is nothing particularly difficult about this process, and having ironed out the practical bugs the biggest problem became how to deal with the larger parts. I reckon the best shape of tank is long and narrow (say, 18 inches long by 3 inches wide, and perhaps 6 inches deep).

You will need the following items:

- A plastic, glass or lead tank to hold the acid bath (all tanks need to be of a size to hold your largest part).
- A plastic or glass tank to hold the dye bath.
- A plastic or glass tank to hold the etching solution.
- A container in which you can boil the parts in water.
- Lengths of pure aluminium rod to suspend the parts in the acid bath (aluminium welding rods are good)
- Sheet aluminium to make the cathode (unless you have a lead tank).
- 12V DC power supply (car battery is OK (2 are even better - see below)
- Set of leads and crocodile clips to make the electrical connections (make sure they are rated for at least 20 Amps, auto jump leads are great for really heavy currents)
- Protective clothing (latex gloves, goggles, apron).

- Somewhere to work! - preferably with a stainless steel sink close by.

The chemicals you will need are:

- Sulphuric acid (10-25% solution, about equivalent to 50:50 battery acid and water if that's your only source).
- Sodium suphate (30 grams per litre of acid solution)
- Sodium hydroxide (lye) at about 10%-20% w/v.
- A water soluble dye (if colouring is desired).

DYES:

The dye is the critical component for coloured finishes. If you are not fussy about exact colour matching then several vegetable-based clothes dyes can be used, I've also had some success with diluted writing inks. The toughest colour to achieve is a truely dense black. Black vegetable dyes produce a gold/brown colour, black inks only produce a dark blue colouration at best. To get a black finish requires the use of a commercial black anodising dye (rather difficult to get hold of in small quantities), or equally effective, a water soluble histological stain called Nigrosin (sometimes called Acid Black 2). You can get the latter from Aldrich Chemical Co., or Sigma Chemical Co. (costs around £20 for 100 grams, £6 for 25 grams - 100 grams is a *lot* of stain, use about 1 teaspoon full per quart of water). Histological dyes can be ordered direct or via your local chemist, or from a store which deals in microscopes and accessories. Other useful histological dyes are Sirius Red, Trypan or Methylene Blue, and there are many other colours should you need them.

REAGENTS:

The acid bath contains 10-25% (V/V) sulphuric acid. Recently, I've been having good success with 10% acid so there probably isn't much need to to higher concentrations. Maybe the 25% will speed the operation up but 25% sulphuric acid is fairly nasty stuff to have lying around in any quantity. A recipe I saw suggested adding about 30 grams/litre of sodium sulphate to the acid bath, but I've had good results without it. If diluting from full-strength (concentrated) acid then you MUST add the acid slowly to the full volume of water, mixing all the time. This is a highly exothermic reaction and the solution will become hot. Never start by adding the water to the full volume of concentrated acid or it may become so hot at first that it may boil and spit acid at you! Sodium hydroxide is available in pellet form, simply dissolve in water to give the 10-20% solution (again, the solution will get quite warm as the pellets dissolve). The active component of many drain cleaners is also sodium hydroxide and will perhaps do as well. Keep the acid and alkali baths well separated! Always wear rubber gloves for all processes to avoid skin contact - including the dye which, if using a histological stain, is particularly difficult to remove.

APPARATUS:

The tanks should be filled to about 2/3 capacity with the relevant solutions. The acid bath requires 2 electrical connections, one is the anode (positive) side which goes to the workpiece to be anodised, the other is to an aluminium plate immersed in the acid which acts as the cathode (negative) terminal. I used some 2" x 1/8" aluminium strip, bent to shape such that it passed down one side of the tank, across the bottom and up the other side. If you are using a lead-lined tank then the lead itself will provide a good cathode. Nothing other than lead or aluminium should be used in the tank. For power I used two 12V auto batteries (55 AHr each) which could be connected either in series (to give 24V) or in parallel to provide high current at 12V. Deep-cycle lead-acid batteries would be better as these are designed to run from fully charged to nearly discharged, ordinary auto batteries should never be allowed to fully discharge. A real problem is to maintain a good electrical contact from the anode to the workpiece. If the part has a tapped hole, then I found the best way was to squeeze the end of the aluminium welding rod (about 3/32" diameter) in the vice to form a spade end, this could then be screwed into the tapped hole. Other than that, the workpiece has to be wrapped tightly with the wire. The problem is that as the oxide layer forms it is possible (if not likely) for it to interfere with the electrical contact - I found that a good initial contact sometimes degraded until little current was being passed. Obviously, take care the workpiece does *not* touch any part of the cathode plate in the tank or it will short the battery.

PREPARATION:

The workpiece to be anodised needs to be clean and completely grease-free. Any fabricated components need to be dismantled, otherwise acid will tend to get trapped in recesses and will cause corrosion at a future date. You can either thoroughly clean the part with a de-greasing agent (alcohol, acetone etc.,) or if the workpiece is freshly machined it can be anodised right away (avoid touching it with the hands). I generally use the sodium hydroxide bath to both clean and etch the surface, 30 mins in the bath will produce a semi-matt finish, 5mins will leave a shiney finish. However, leave it too long and the whole thing might dissolve. Wash under the tap before placing in the acid bath. To form the oxide layer will require about 1.5Amps per 3-1/2 square inches of surface area. So, a large-ish part 6 inches square would need about 20 Amps - a high current!! Think about that before dunking your engine block in a big tank! Note: the process works best at lower temperatures, 'hard' anodising is done at 3 deg C. Putting 20 Amps or so through the bath tends to heat it considerably.

ANODISING:

OK., you have the part suspended in the acid bath using aluminium wire, and the cathode is connected to the negative terminal of the battery. I use no current control, and a part of any given size will draw a current as indicated above. There will be (should be) fine bubbles released from the cathode during the process, and the workpiece will take on a milky-grey appearance as the oxide layer forms. It will take 70 minutes to form a layer of the correct thickness for black dying, about half that for a decorative coloured finish.

IMPORTANT NOTE: If the part has an etched or otherwise rough/matted finish then the effective surface area may be UP TO TWICE that which you may have calculated from the gross overall dimensions. Take this into account when estimating the total time for the electrolytic formation of the oxide layer.

After the time is up it's merely necessary to remove the workpiece from the acid bath, rinse it under the tap to remove excess acid, and dunk it into the cold dye bath. Some sources suggest you might need to boil the dye bath but it works fine for me cold. Of course, you don't *have* to dye the anodised part - clear anodising still offers good protection from corrosion. Leave the part in the dye for about 30 minutes then hook it out and rinse it under the tap. The part should still retain the colour after the rinse (if not, something's gone seriously wrong somewhere - probably you lost the electrical connection part way through). After rinsing place the the part into a pan of boiling water for about 30 minutes to permanently seal the surface.

And that's it - you can add a little more protection to the finish by polishing with wax or lanolin. Pictures below are of my own setup for anodising small parts (to about 6 inches square).

(Addendum: Further experiments revealed several pesky bits of alloy of unknown parentage which firmly rejected the cold dying process. I resorted to boiling the dye bath which then worked very well. The practical problems of dealing with boiling black dye are real - this is not the stuff to spill all over the kitchen cooker (take my word for it - the dye solution boils over like milk).

After several failures due to loss of electrical connection to the workpiece, my standard method now is to drill a 3/32" hole and twist in a length of 3/32" alluminium welding rod. This connection never fails and always results in a good finish. Further, I make use of my multimeter connected between power supply and workpiece set to measure current (it has a range up to 20 Amps).

Temperature of the acid bath is important, in as much that it should not be allowed to rise above about 25 deg C. Some large pieces (requiring 20 AMPs) heated the bath to a temperature of almost 50 deg C, and the dye failed to produce an even colour. Having invested quite some time on these workpieces I figured it worthwhile trying to strip off the failed anodised surface in the NaOH bath. I only had partial success (probably didn't leave them in the bath long enough) as repeating the anodising process only resulted in a two-tone black finish. Anyway, my main acid bath now sits inside a larger tank which I fill with water and ice to keep the temperature down.

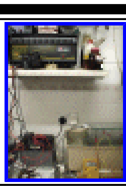
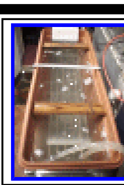
Addendum II: I finally gave up re-charging batteries and bought a heavy duty 180 AMP battery charger/starter. This unit will run at up to 180 AMPs continuously and posses ample output to cope with any size anodising job I'm likely to tackle. It was quite cheap (I thought) at just 49UKP (German manufacture from Halfords).

Addendum III: I've been searching around for suitable containers for larger workpieces and eventually spotted a useful item in the form of a 2ft x 6" x 6" plastic box designed for growing plants in. I bought four and used two as covers for the other two. These only cost about 2 UKP each from garden supplies shops.
Another note on cooling: now that I can anodise larger pieces cooling became even more critical with the increase in current being used. It was not practical to use a two bath arrangement so instead I ran coils of plastic tubing (about

3/8" diameter) around the bottom of the bath, with a constant flow of cold water running through it to remove excess heat. The method works very well. (Caveat) - the method works well provided the plastic tubes don't split :((The picture below showing the big tank also shows the series of aluminium tubes connected together forming the cooling grid. I failed to round-off the sharp edges with the result that the silicone tube eventually split at that point. This resulted in 2 gallons of sulphuric acid overflowing onto the workshop floor while I wasn't there watching it. Moral: round of the tube ends, and arrange an overflow so that if this should happen the acid is safely directed to a waste. Ah well, I got a clean floor anyway...

The major problem with anodising large(-ish) parts is that the apparent probability of having a surface defect appear on the finished part increases exponentially with the larger surface area (!) Small surface blemishes (pin-holes in particular) which might go un-noticed beforehand show up as noticeable white doughnuts on the dyed part. It's almost impossible to anodise the suface of aluminium barstock - even if you thoroughly clean it with emery paper, you really need to get 10-15 thou under the surface to expose new metal. Even thorough etching (20 mins) in NaOH often fails to prevent 'doughnuts' appearing on the stock finish, probably because oil or grease (or hard resinous stuff) trapped in the pinholes prevents the etching solution getting in. I have some tips which may help if you are having problems attaining a rich, dense black, shiney finish (possibly the most difficult to obtain):

- *Be scrupulously careful not to handle the part with bare hands at any stage - I've had one part come out of the dye bath with a perfect rendition of my fingerprint!*
- *Clean all traces of acid off the part before placing in the dye bath. Look particularly for signs of crystals forming on the surface during the electrolytic procedure (this will prevent formation of an evenly distributed oxide layer), a sure sign that the acid is becoming saturated with aluminium sulphate. The only answer here is to replace the electrolyte with fresh acid.*
- *You can leave the part in the dye bath much longer, I've left parts soaking up to 3 days with good results. The addition of 5-10% alcohol (ethyl or propyl) to the dye bath will also help penetration of the dye, but BE WARNED - this makes the dye bath a potential fire hazard, especially so if you tend to heat it.*
- *The parts in the dye bath must be seperate from each other, and also the sides of the bath. Ideally, each part should be suspended in the dye solution, and the solution agitated to disperse any suface bubbles that may form on the part (yet another sure way of getting white spots).*
- *Similar recommendations apply to the hardening water bath, keep the parts suspended clear of other objects. You can leave the parts boiling for several hours with no harm as once the crystalline surface is formed the dye cannot leach out into the water bath.*
- *A good way of polishing the surface is to use T-cut (or similar), a proprietary cutting compound for finishing auto paint finishes. This is not as abraisive as metal polishes and produces a beautiful shine with reduced danger of removing the black anodising on sharp corners.*
- *Having been anodising on a semi-commercial basis for over a year now (making aluminium telescope parts) I can add that it's very important that the dye bath be vigorously agitated to achieve a dense, even colour without surface pitting. It seems that some alloys have inclusions which lead to small pits forming whilst immersed in the acid bath, and these small pits turn into very large pits if the parts are just left standing in the dye bath. Agitation actively washes away any residual corrosive residues allowing the dye to penetrate better and also preventing the 'white spots' that would otherwise spoil the finish. This is particularly true for rough, matt or complex surfaces (e.g., a knurled finish). For agitation I use a small circulatng pump.*

 180 Amp Power supply (upper left).	 Small acid bath and connectors. Etching bath on the right.	 Large (2ft) acid bath made from plastic trough. Note water cooling grid.

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