SEALING ANODIZED ALUMINIUM—RECENT DEVELOPMENT

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Sealing of anodized aluminium has been highlighted, need focussed and recent developments in the seventies reviewed with reference to their importance, principles, mechanism, process techniques-equipment, prescaling. operating conditions, economics and future trends. Some guidelines and typical sealing processes in tabular form are given from the practical view point. Pertinent reference are cited to enable ease of access to original literature.

A large and fast growing fraction of aluminium is anodized for aesthetic appeal, hardness and erosion resistance, preventing attack on the substrate, as well as for applications where electrical insulation is of importance. This fraction can be estimated as at least 12% of the total world production of 8,500,000 tons[1]. The aluminium just after anodizing, especially from acid electrolytes like sulphuric acid, has a porous and chemically active film. It is therefore of little commercial value. The primary object in sealing is to convert this anodic coating to a stable, unreactive chemical state so that the conditions of environmental exposure will not adversely affect the appearance and integrity of the coating. Efficient sealing is very important where long life, maximum corrosion resistance, and preservation of colour are called for. However, improper sealing leads to discoloration and dirt collection with diminished aesthetic.

Though the process of anodizing has made rapid advances and the output of literature is quite vast, sealing part has been neglected and mentioned briefly as an ending touch. Only in recent years, relatively more studies have been made and a variety of sealing processes are currently available for commercial use. Most of the work is, however, covered by patents. This paper outlines the developments in the seventies in this field. The objective is to present an elementary view of the present and future of this technology, with the specific aim of providing an introduction and focussing the 'need' for the process. Several reviews have been published on various aspects of sealing[2–15], as well as a few detailed ones covering integral colour[16], architectural[17] and hard [18,19] anodizing. A few books[20–25] on anodizing of aluminium, covering this topic are also available.

Principles and Mechanism

When aluminium surface is made the anode in a suitable electrolyte like sulphuric, oxalic or malonic acid, an oxide film of controllable thickness and quality can be formed on the surface. However, the resulting film consists of a thin compact "barrier layer" adjacent to the metal and a thicker outer porous layer. This porous layer has a large surface area per unit of projected area, because it consists of nearly hexagonally close-packed cylindrical pore normal to the metal surface and extending to the outer surface. The spacing and diameter of the pores depend on the conditions of formation. The high surface area on account of this high porosity makes the surface chemically very active.

The process of sealing consists of reducing the porosity of the anodic film due to a reaction between the oxide and the sealing medium. Sealings of the porous films are normally by heating them in media such as water, steam, certain salt solutions or treatment with waxes and lacquers. With sealing, anodized films undergo reduction in porosity, change in crystal structure, lose the ability to absorb dyes, and show improved corrosion resistance. During the hydration treatment, the oxide film gains in weight, the pores are pressed together to fill up and close completely if boiling is continued long enough. Thus sealing smoothens the surface and makes it impervious to soil[15].

As regards the MECHANISM of sealing, very little is clearly understood about the nature and structure of sealed film. The most successful tools for investigating the sealing of anodic coatings on aluminium have been reported to be the study of N.M.R. and Infrared Spectroscopy[26]. Two types of tests used to describe the quality of seal are electrolytic resistance (non-destructive) tests and acid dissolution tests. The former includes reflectance and admittance tests while the three major tests of the latter are Modified Kape test, Chromic-Phosphoric test and ISO Acetic acid dissolution test[27]. Various other techniques have been used to illustrate the mechanism and quality of

sealing. These include Impedence test[28], Electrical sealing tests, Coulometric Method, and effluent gas detection (EGD) method[4,15]. All these studies show that sealing process either wholly or partially blocks the pores in the anodic film[29]. Also the crystallites increase in size ("swell up") and fill up or obliterate the pore structure. In general, two types of reactions take place during sealing.

(a) formation of aluminium oxide monohydrate or BOEHMITE as per the following reaction :

$$Al_2O_3 + H_2O = 2\ AlO(OH)$$

This is a favourable process thermodynamically at 25°C with G = −1.54 K Cal./ mole Al_2O_3. Pore filling is reported[82] to proceed mainly by inward movement of the pore wells, caused by the less densa AlO(OH), the formation of which has been confirmed by the Infrared spectra and is believed to be precipitated near the pore mouths in very small concentrations[26].

b) formation of aluminium oxide trihydrate with a structure analogous to BAYERITE.

Conditions of sealing, such as temperature, pH, time of sealing and the presence of impurities are some of the principal factors in determining which of these reactions predominate during the sealing step[15]. Chromate solutions are reported[30] to give more rapid sealing than the commonly used nickel acetate sealing, whereas plain water gives much slower sealing. The physicochemical mechanisms of sealing are also reviewed[31].

The Process

Equipment :

The sealing tanks should be of stainless steel[32] or other inert material like Monel. Rubber or lead-lined steel can also be used, while for simple water rinses, tanks made of reinforced concrete, lead-rubber-plastic lined mild steel, galvanized mild steel (not suitable for acid rinses) or wood (fitted with weir or standing overflow), acid resistant brick, P V C or Fibreglass have been mentioned[21].

Pre - Sealing :

Before sealing the anodic film, any masking material used in previous processes must be removed. Likewise, any residual acid must be removed. It has been reported[33] that there is tendency to produce cloudiness in bright anodizing with about 100 ppm of sulphate in a sealer. Also for coloured effects, the anodized aluminium may be immersed in dye solutions before the film is sealed. Different methods, including electrolytic, have been used to remove the residual electrolyte retained in the pores of the anodic coating. These are in addition to simple rinses.

For example, one method comprises electrolytically treating anodized aluminium articles in an aqueous alkaline solution containing one or more ammonium salts of chromic acid, dichromic acid, phosphoric acid, silicic acid and metasilicic acid and an amine or ammonia ; the treatment carried out by applying a.c. or d.c. while changing the polarity of the electrodes, below 60°C and 3–70 V for 2 seconds to 2 minutes. The amine used in the method is dimethylenediamine, triethylamine, ethylene diamine, hexamethylene diamine, monoethanolamine, dimethylaminoethanol, isopropylamine, triethanolamine and morpholine[34]. Another similar process[35] is patented in which an aqueous solution containing amine salts of high molecular thermosetting or thermoplastic resins like alkyd melamine, acrylic and epoxy resins are used ; the electrolysis being carried out at less than 100V for 3 to 5 minutes. The coated surface is then baked at 200°C for 5 minutes. Another patent[36] discloses presealing at 15–100°C with solutions containing dextrin (0.1 to 5g/l) with viscosity of 50-400 cp.

Sealing :

Table I gives details of some typical sealing processes that are used after anodizing aluminium and its alloys.

Basically, there are two types of sealing :

i) **Physical Sealing** : Consists of plugging the pores with organic materials like oils, waxes, greases, lacquers, and silicones. There is no swelling of the surface and the process has certain advantages both as replacement and as an addition to the chemical sealing. For example, physical sealing is preferable for infrared reflectors, as the chemical sealing has poor infrared reflectivity[21]. However, for corrosion resistance, physical sealing is not as good as chemical one, although some lacquers are quite corrosion resistant and have a high dielectric strength[40].

ii) **Chemical Sealing** : is the controlled hydration of anodic film. The term "Sealing" is normally referred to chemical sealing only. It is necessary and in fact universally applied when maximum corrosion resistance is desired. It causes the

Table 1 : Typical Sealing Process

	Composition	pH	Temp.	Time	Remarks	Ref.
1.	Water, preferably distilled or demineralized.	5.5 (adjusted with acetic acid or caustic soda)	Not less than 95°C	As anodising cycle	Widely employed. Reliable with good opreating conditions, but less so otherwise. Some dyestuff may bleed.	37
2.	Saturated Steam	—	100°C	15-45 min	Very efficient and clean, avoids bleeding of dyestuff, but relatively expensive in capital outlay. Not in extensive use	37
3.	5g/l Nickel acetate buffered with 5g/l boric acid (preferably distilled or demineralized water)	5.6 to 5.8	95°C	3-30 min	Sometimes short immersion is followed by standard water seal to avoid 'bloom'. Can improve fastness of some dyes, but may be detrimental to others.	37
ii)	5g/l Nickel acetate and 2g/l n-ethyl-butyrate	—	95°C	10 min	Passes sealing, alkaline and acid test, but conventional sealing by boiling is unacceptable.	38
iii)	5g/l Nickel acetate + 5g/l boric acid + 1 ml/l melting agent	5-3 to 5.5 (adjust with acetic acid)	93.5°C	—	Use of filter enables a number of colors to be sealed in the same bath.	32
4.	20-50g/l sodium or potassim dichromate	6	80-100°C	10-30 min	Produces yellow discoloration. Widely employed for service and aircraft specifications.	37
ii)	100g/l potassium dichromate, 18g/l calcined soda or 13g/l caustic soda	6-7	90-95°C	2-4, but up to 10 min.	Corrosion resistant and rapid.	39
iii)	15g/l Potassium dichromate + 3g/l caustic soda or 4g/l calcined soda	6.5 to 7.5	90-95°C	same as above	Carbonate interferes each addition of soda to be followed by boiling to remove carbon dioxide.	39
5.	Lacquers	—	air dried or stoved	—	Usefull for temporary protection.	37
ii)	Paraffin wax, lanolin, ceresin wax, boiling linseed oil, varnishes		Drying		No swelling.	39
6.	5g/l Sodium Silicate		90°-100°C	30 min.	Occasionally used, gives good protection against weak alkaline solutions.	15

swelling and constricts the pores, resulting in the closing of pores in the anodic film. The following are the major chemical sealing processes :

a) **Hot Water Sealing** is most widely used process and typical process is given in Table 1. Although a slow process, there is no chemical reaction with the dye. The critical factors in this operation are the temperature, pH and the purity of water.[21]. It has been claimed that the rate of sealing may be increased by adding surface active agents like assymetric dimethyl or ethylene diamine hydrochloride[15].

b) **Steam sealing** is rarely used as it is more expensive. However, it avoids solution contamination and is preferred on cold-dyed multicoloured work as there is no bleeding of the dyestuff. A typical steam sealing step is given in Table I.

c) **Sodium silicate sealing** is also employed occasionally, the usual procedure being to heat in a 5% sodium silicate solution. This gives good protection against weak alkaline solutions[15]. Typical process is given in Table I.

d) **Dichromate sealing** has become very popular for non-decorative purposes, as it gives much superior resistance to corrosion. Dyed coatings with the exception of yellows cannot be sealed by this method. In dichromate sealing, the work should be thoroughly rinsed in hot water to remove any excess of sealing solution. While simple hot water develops corrosion spots, this dichromate sealing prevents it[35]. Typical sealing with dichromate is given in Table I.

e) **Metal Salt Hydrolysis sealing** is very popular and in this process, the solutions of various salts are absorbed into the porous coatings where they are hydrolysed and precipitate hydroxides. Substances which have been suggested include the acetates of nickel, cobalt, cadmium, zinc, copper, aluminium, lead, sodium and barium and certain sulphates, oxalates, chlorides, nitrates, citrates, tartarates, fluorides and other miscellaneous compounds[15].

f) **Nickel-Cobalt Acetate sealing**, along with hot water sealing, accounts for nearly all the aluminium anodized and dyed and is of considerable practical importance. It is claimed that nickel acetate type of solution is less affected by such contaminants as phosphates (10 ppm tolerable), fluorides (15 ppm tolerable) and silicates which markedly reduce hydration in case of simple hot water sealing. Also addition of certain anionic type wetting agents to nickel acetate greatly reduces smudge. All salt sealing processes are followed by at least a hot water rinse. In general, a final seal in deionized water is desirable[21].

g) **Lead acetate[41] sealing** and more recently sealing in **Zinc, Copper, and Chromium Salts**[42] improves the light fastness and corrosion resistance to heat and atmosphere in the case of certain dyestuffs which are not effective by nickel/cobalt acetate treatment. Lead acetate sealing is more suitable with various aluminium red dyestuffs.

Recent Developments

Two processes which claim to prevent sealing bloom, have been patented, one using solution of acrylic, methacrylic or maleic acid in 0.7% solution in 2N sodium hydroxide at 20°C before or combined with sealing[43] and the other requires addition of amine, Schiff's base or ammonia or (b) dextrin or lignin sulphonic acid[44] in the sealing solution. Two Japanese patents cover the sealing in solutions containing hexamethylene tetramine or mono or triethanolamine and aminopolycarboxylic acid[45]. Another patent[40] discloses addition of alkyl naphthalene sulphonic acid, sodium lauryl sulphonate and condensation product of acetylphenol with ethylene oxide to the nickel and or cobalt acetate sealing solution. Bleeding of colours, especially copper red colour, is claimed[46] to be prevented by addition of 0.1 to 2 g/l gelatin in sealing with hot water. Aqueous sealing solutions of tungstate and plumbic acid with amines[47], dichromate with vanadate[48], sodium-potassium or ammonium molybdate[42, 49, 50] neutral sodium sulphate solution[51], concentrated sodium, potassium or calcium sulphate or chloride solutions[52], chromic acid and a complex fluoride compound[53] solutions have all been patented.

Corrosion resistance of anodized aluminium is claimed to be improved without affecting appearance, by sealing in fused bath of aliphatic compound such as

lauryl stearate, stearic acid or paraffin at least at 150°C The water in the coating evaporates in the molten material and the pores are filled with fine particles of hydrocarbon[54].

During 1972, nickel and cobalt sealers with organic additions [40,55] were patented while in 1973, sealing accounted for two patents, one to Yoshimara[56] for use of an oxygen acid ion of phosphorous and second to Cohn[57] for solution containing disodium 4-dodecylated oxydibenzene sulphonate and a metal salt, in which a dye could be included. Also a new product[58] for the bloom-free hot water sealing is claimed in the same year. In 1974, developments have been reported exclusively by inventors. New sealing processes have been patented by Wallace[59] for using triethanolamine followed by desmutting in a mineral acid and by Friedemann and Germscheid[60] for employing a hydroxycarboxylic acid. Other claims include a clear sealer[61] comprising chromate and tungstate while metal salt[62] and a dual process[63,64] are also in metal salt sealing. In one patent[65], the oxide film is impregnated with cobalt ions, then treated with a chromate solution whereas electrolysis in a calcium and magnesium salt bath[66] is disclosed in another. In 1975 also, three patents concern sealing. The use of a suitable dye in the sealer is reported to neutralize any undesirable colour due to the alloy[67]. The second patent discloses sealing in a nickel or cobalt acetate, then desmudging in an acid bath[68] while the third claims a solution of calcium ion and a phosphate for sealing[69].

Guidelines for Successful Sealing

It must be clearly understood that good sealing improves corrosion resistance and preserves the appearance whereas improper sealing spoils the anodized film. Therefore, utmost care has to be taken in sealing these anodized aluminium or its alloys. The critical factors in this operation are the pH, temperature (especially in nickel acetate-sodium dichromate or duplex sealing[70], time of sealing, purity of water and composition of sealing solution, bath agitation etc. In general, the best sealed films have the lowest conductance.

For successful sealing, some of the guidelines are given below :

1. Before sealing, rinse the anodized articles thoroughly in running water to remove any residual acid.
2. Remove any masking material used in earlier processes before sealing.
3. Adjust the pH to the desired value with acetic acid etc ; however, too low a pH causes leaching out of the dye[32].
4. Use preferably distilled water for sealing, as tap water and even deionized water in some cases, may lead to water stains and inferior corrosion resistance[21].
5. Add 0.0.% potassium dichromate to water if chlorides are present in harmful amounts[21].
6. Use steam sealing where solution contaminations are predominant, also on cold-dyed multicoloured work[21].
7. Use filter where a number of colours are to be sealed in the same bath[32].
8. In automatic plants, use counterflow rinsing and ion exchange column in recirculation with sealing water maintaining sulphuric acid[71] below 5mg/l.
9. Specify testing of sealing to BS 1615 or DIN 50018.
10. Do not seal hard anodic coatings, as sealing can reduce abrasion resistance by 10-20 per cent[18] However, these can be lightly waxed.
11. Seal for at least 25 minutes where the anodizing time is 30 minutes[28]
12. Rinse thoroughly in hot running water after sealing.

Future Trends

The developments in sealing of anodized aluminium during the seventies have been quite marked though not spectacular. Conventional hot water and nickel acetate sealing continue to hold the field as the basic solution and the changes in this respect are by way of addition to these.

The sealing of anodized aluminium at room temperature is quite attractive, and may also prove less expensive. Such a process which uses sodium sulfantimoniate, has already been patented. By the same process, but at 40°C, a bronze-yellow colour is reported to be obtained[72]. Another process[29] is stated to seal the pores in cold solution, without causing hydration and using tannin (5g/l). Similarly an unusual black dye[73] for anodized aluminium is available, which is reported to be self sealing, has good light-fastness and smutless and eliminates a separate sealing tank and rinse. Developing countries like India, where the cost

of sealing sometimes exceeds that of anodizing, would no doubt be interested in such processes for economy and quality.

In future, the variety of commercial processes are expected to grow. Conventional pure water sealing may be a thing of the past. Novel electrochemical processes will be used to improve corrosion resistance permit automatic process control and make it less expensive. It will also mean the rapid and intelligent adoption of improved processes which are already developed. It is concluded that the future developments in this field will be directed towards modified or new types of baths, better inventory control and improved techniques, so that sealing process can become fairly economical.

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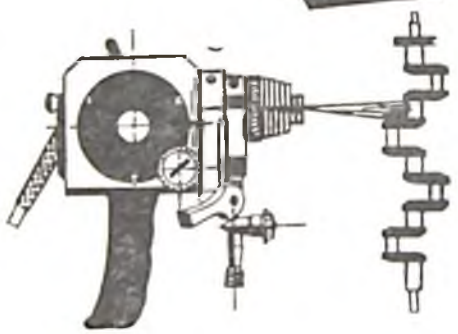
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