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## 1 <u>PRE-TREATMENT</u>

Before the powder coating is applied on any substrate, it is advisable to undergo pre-treatment in order to enhance the durability of the powder coating. Pre-treatment methods of metal substrate prior to powder coating are divided into 2 type, phosphating and chromating. Each type of pre-treatment is then sub-divided into another 2 two types.



#### 1.1 PHOSPHATING

Also known as conversion coating, phosphating is an application of an iron or zinc phosphate coating to the substrate. This conversion coating of pre-treatment process is a crucial process as it significantly increases the performance of the finished coating.

A conversion coating which is developed on metal substrate, either ferrous or non-ferrous surfaces, converts the metal substrate to a uniform, inert surface. This process increase the overall resistance against corrosion of the finished part, improves the bonding and minimizing the oxidation on the scratched part of the coating.

Parts are subjected to an acidic bath and a chemical conversion forms a complete film on the part surface, changing the chemical and physical nature of the metal surface. The coating that will develop on the surface of metal can be iron, zinc, polycrystalline, chromate, or manganese phosphate film. OXYPLAST

OXYHIN SDN. BHD. (393471-X)

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## 1.1.1 Iron Phosphating

Iron phosphate is the thinnest phosphate films. During the phosphating process, a flat or amorphous metal phosphate topcoat will be formed on the firstly developed iron oxide base. Once treated, the metal surface will have a blue iridescent or blue-gold iridescent colour, depending on the coating weight and the base metal. A typical iron phosphating process consists of:

- phosphate acid base
- accelerators/oxidizers
- surfactant package (optional)

In an iron phosphate solution, some iron ions are released into the solution when the metal surface is etched, producing a positively charged surface. The iron ions in the solution will be converted into iron phosphate, negatively charged. A pH rise occurs at the interface of the solution and the part, causing the iron phosphate ions to deposit an amorphous coating on the metal surface. Nitrite or chlorate, as oxidizers, initiate attack on ferrous parts to produce iron for the iron phosphate coating. Accelerators, such as molybdate or vanadate, provide active sites for iron phosphate deposition. Choice of oxidizer of accelerator in a particular product may affect the performance or appearance of the final coating.

A cleaner/coater combination followed by a rinse is the typical minimum chemical cleaning and phosphating process used. The number and type of process stages is dependent on finished part requirements, thus the addition of stages in the process can provide enhanced performance. The multi-stage spray washers are the frequently used methods which consist of as few as two stages and as many as eight.

- Two Stage: clean/coat, rinse
- Three Stage: clean/coat, rinse, rinse/seal
- Four Stage: clean/coat, rinse, rinse/seal, DI rinse\*
- Five Stage: clean, rinse, phosphate, rinse, rinse/seal
- Six Stage: clean, rinse, phosphate, rinse, rinse/seal, DI rinse
- Seven Stage: clean, clean, rinse, phosphate, rinse, rinse/seal, DI rinse
- Eight Stage: clean, rinse, clean, rinse, phosphate, rinse, rinse/seal, DI rinse

\* Deionised water; water that has been filtered to remove negative and positive ions



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## 1.1.2 Zinc Phosphating

Zinc phosphate is a non-metallic, crystalline coating that chemically adheres to the substrate. Being very adherent, zinc phosphate form an uniform coating which increase the coating adhesion properties, and giving better coating in recessed areas along with better corrosion resistance. Typical zinc phosphate consists of:

- phosphoric acid base
- accelerators
- zinc salts

Unlike iron phosphate which comes from the part surface, zinc phosphate comes solely from the mixture solution itself. On the part surface, crystals begin to form at anodic sites and stop forming when they hit another crystal. The more origination sites the better the density of the coating. For powder coating, it is best to keep the structure orderly and densely packed. Powder does not stay in the flow stage for very long. Larger phosphate crystals may not allow the powder material to completely wet the surface and a capillary layer may form under the coating. Moisture will penetrate the coating and cause corrosion that will lift the coating from the surface. A zinc phosphate can not clean and coat simultaneously in a three-stage process like iron phosphate, a separate cleaning stage is required.

# 1.2 <u>CHROMATING</u>

Chromating is the most used conversion coating process for aluminium profiles which are going to be powder coated. The chromate layer enhances the adhesion between the metal surface and the lacquered product. If chromate layer is thick, it will give a very good corrosion resistance, but the adhesion will be poorer compared to a thinner layer. However, chromate layer which is too thin will result in good adhesion, but poorer corrosion resistance.

A general chromating process prior to powder coating will follow a sequence of steps such as below:

- 1) degreasing
- 2) rinsing
- 3) etching
- 4) rinsing
- 5) deoxidisation
- 6) rinsing (double rinsing)



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7) chromating
8) rinsing (double rinsing)
9) drying
10) powder coating
11) curing

The process of chromating and powder coating is shown in Figure 1 below:



Figure 1: Process of Chromating and Powder Coating

# 1.2.1 PROCESS OF CHROMATING

### 1.2.1.1 DEGREASING

Grease, oil and other surface contamination will be removed from the substrate surface in this degreasing step before the substrate undergone etching process. In order to protect the surface from being attack during this cleaning process, often inhibitors are added. The cleaning is done under temperatures between 50°C and 70°C while the duration is about 3 to 4 minutes and higher depending on the surface conditions.

### 1.2.1.2 <u>RINSING</u>

To facilitate the removal of chemicals from the substrate surface and prevent contamination of solution being carried over, there are usually one or more rinsing steps after each process step. Mains water is normally good enough after the first pre-treatment steps, but in the final rinsing step the water should be very clean to avoid adhesion and corrosion problems. In this case, the conductivity of the rinsing water (de-ionised water) should not exceed  $30\mu$ S/cm. It is crucial to use the de-ionised water to do the rinsing, as salts (calcium) caused by hard water dried on the extrusion surface can be the starting point of corrosion and lacquer blistering. There are no limits regarding the rinsing steps after chromating should not exceed  $50^{\circ}$ C. The chromate layer could be washed off if the



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water is too hot. Rinsing duration is generally few minutes and it is and advantage to get better rinsing by agitation of the jig (profiles) and/or air agitation of the water.

### 1.2.1.3 ETCHING

The purpose of etching process is to remove the thin natural oxide layer on the surface before the chromating. Inhibitors are also often added to protect the metal surface from attack during the etching process. The etching process is done under temperatures between  $50^{\circ}$ C and  $70^{\circ}$ C while the duration is about 3 to 4 minutes and higher depending on the surface conditions.

### 1.2.1.4 DEOXIDISATION

In the deoxidisation process, any smut resulting from insolubles from the etching is removed. This smut layer consists of oxides like magnesium oxide, intermetallics, silicon, etc. Usually a dip in an acid solution such as nitric acid or sulphuric acid will remove the smut, however, special deoxidisation products from chemical suppliers is used (in addition to nitric acid) by many producers of coated aluminium profiles. These deoxidisation products may consist of chromate in order to inhibit the reoxidisation process on the aluminium surface, and fluorides to remove silicon. Depending on the smut layer, the solution and the alloy, a dipping process will consume from about 30 seconds up to 5 minutes under normal room temperature.

#### 1.2.1.5 GREEN CHROMATING

Phosphochromating or green chromating process is carried out in a phosphochromate baths. The main components of the bath are phosphoric acid and/or an acid phosphate, hydrofluoric and chromic acid or another source of hexavalent chromium. The pH of the bath is maintained, usually at about 1.7 to 1.9 but sometimes lower. This can be achieved by adding phosphoric acid and a buffer component. The temperature in the bath should be between  $25^{\circ}$ C and  $30^{\circ}$ C.

Investigation of a phosphochromate coating shows that the bulk of the coating consists of a hydrated from of chromium phosphate with smaller amounts of chromium oxide present. As the aluminium oxides and fluorides present in the intermediate surface regions, the chromium oxides are concentrated towards the aluminium surface.

The phosphochromate conversion coatings are green in colour. As the coating weight increases from 0.4 to  $1.2 \text{ g/m}^2$ , the intensity of the colour changes from iridescent



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green to dark green. The coating should be as even as possible, adhere to the substrate and free from powder. Nevertheless, the colour and its uniformity may differ through visual examination, depending on the material and its surface conditions. The structure of green chromating is shown below.

### 1.2.1.6 YELLOW CHROMATING

This chromating process can be done in either unaccelerated or accelerated chromate baths. The unaccelerated chromate baths are essentially composed of chromium (III) oxides (another source of chromium ions) and of hydrogen fluorides (complex hydrofluoric acids, or their salts). To obtain the desired pH at about 1.8 to 2.1, nitric acid is usually added in. Other substances are present in appropriated formulations to achieve better conditions in the industrial use. The temperature of the bath should be around  $25^{\circ}$ C. In the accelerated baths, potassium ferricyanide is usually added, but many other variations have been developed. Cyanides are less used due to environmental concern.

From the examination of the chromate layer, it is known that a thin outer layer consisting of chromium ferricyanide and hydrated chromium oxide conceals the bulk of hydrated chromium oxide coating. Small amount of aluminium oxide and fluoride too are present in the interface between the film and the aluminium.

A yellow layer is obtained from this chromate conversion coating, with a variation of intensity from iridescent yellow to golden tan as the coating weight increases from 0.4 to 1.0 g/m<sup>2</sup>. The limit value of 1.0 g/m<sup>2</sup> has been established because of powdering phenomenon of the conversion coating if exceeded. The structure of yellow chromate pre-treatment is shown as below.



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## 1.2.1.7 DRYING

After the chromating and rinsing process, prior to powder coating, the profile has to be completely dried. The coating process will be interfered if moisture presents on the surface. The temperature of the profile should not be too high in the drying oven. The breakdown temperature for the green chromate coating and yellow chromate coating are about 80°C and about 65°C respectively. Poorer corrosion resistance will be obtained if the drying temperature exceeded these limits.

### **QUALICOAT** aluminium pretreatment:

<u>Chromatation</u>: (150-1200 mg/m<sup>2</sup>, but take into account that Qualicoat specifies 600-1200 mg/m<sup>2</sup>)

Basically the (yellow) chromatation by immersion concerns the following steps (source: MAVOM, a Dutch institute for metal pretreatment):

- 1) degreasing
- 2) rinsing
- 3) etching
- 4) rinsing
- 5) rinsing
- 6) Cr-VI chromatation
- 7) rinsing
- 8) demi rinsing

Non-chromate pretreatment: (100-150 mg/m<sup>2</sup>)

The same MAVOM institute applies a Al-Zirconium-Titanate pretreatment as chrome-free variant:

- 1) degreasing
- 2) rinsing
- 3) etching
- 4) rinsing
- 5) demi rinsing
- 6) chrome-free conversion
- 7) demi treatment (nebulized)