HOT-DIP COATINGS: A REVIEW

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Of the various types of coating, hot dipping is one of the oldest methods of coating one metal with another and is still in wide use today as a means of producing an inexpensive, protective film. The coating is formed from the metal which adheres to an article when it is dipped in a molten metal bath. Metals used commercially for such coatings are limited to those of low melting point to avoid thermal changes of the basis metal. Zinc, aluminium, tin and lead account for most of the dipped coatings produced.

Of the common hot dip coating metals, zinc and aluminium are anodic to iron and hence offer galvanic protection to iron in most environments. Lead and tin are cathodic to iron and rely on the barrier film principle to provide protection.

Hot dipping with reference to iron and steel as the basis metal is only considered in this review. This is because iron and steel in its many forms are most universally used in greater proportions than other metals and alloys

CLEANING :

In order to form a uniform, adherent coating the work must present an uncontaminated surface to the molten metal. Therefore, degreasing is the first step in hot dipping which remove soils, grease and oil sticking with the materials, carried out with an organic solvent or a hot alkaline solution. The degreasing method varies with the type of soil to be removed. Vapour degreasing, solvent cleaning, and emulsion cleaning are often satisfactory when only light oil must be removed, but hot alkaline cleaning is more versatile and does a more thorough job in removing grease and drawing lubricants. In certain cases electrolytic cleaning is adopted for the removal of rolling lubricants from cold rolled steel.

Following the degreasing treatment the work is well rinsed with water and then pickled in acid to remove oxide scale. Various acids are used for steel pickling, including sulphuric, hydrochloric and phosphoric acids. Table I shows typical operating conditions for pickling

Table I Typical Pickling Baths

Bath characteristics	Sulphuric acid	hydrochloric acid
Concentration, % by wt	5—15	8—12
Temperature, °C	60—76	38—41
Immersion time, min	5—20	5—15

in sulphuric and hydrochloric acid bath. Excessive attack on the basis metal during pickling is prevented by the addition of a suitable inhibitor, such as an amine. The cleaning operation in many cases is done by abrasive blasting avoiding degreasing and pickling. Abrasive blast cleaning entails the forceful direction of abrasive particles —either dry or suspended in a liquid against the surfaces of metal parts or products, to remove contaminants. The typical uses of abrasive blasting include a) removel of rust, scale, dry soils, mould sand or paint b) Roughening of the surfaces in preparation for bonding or coating (c) removal of burrs, and (d) development of a matte surface finish. The materials used in dry abrasive blast cleaning may be categorised as grit, shot, sand and miscellaneous. In wet blasting very fine particles are used which include silica, quartz, aluminum oxide, other refractory abrasives and bright shot (glass beads). Eventhough steel sheets and articles are cleaned by blasting technique without any pickling at all, it is more usual to give a quick acid dip.

FLUXING :

Although degreasing, pickling, water rinsing, and other cleaning procedures remove most of the surface contamination and scale from iron and steel, small amounts of impurities in the form of oxides, chlorides, sulphates and sulphides are retained; unless removed, these impurities interfere with iron and coating metal reaction when iron or steel part is immersed in coating metal. A flux is used to remove these impurities and to counteract oxidation of the basis metal as it enters the molten metal bath. The flux most commonly used is zinc ammonium chloride, $ZnCl_2$ $3NH_4Cl$, which at the bath temperature, not only cleans the metal surface but also iron salts which may have been carried over from the pickling bath.

Coating procedures for the various metals and the specific properties of the interface are described separately below:

GALVANIZING :

Galvanizing is, perhaps, the most important of all hot dip coatings[1-10] but this review excludes galvanizing from discussion here and deals with other coatings only.

ALUMINIZING :

Steel strips are commercially aluminized [11,12] by first oxidizing the strip in a furnace maintained at 870°C. It is then exposed to a controlled reducing atmosphere when the uniform oxide layer is reduced to metallic

iron. The work is cooled to 700°C, then introduced into the bath where the iron coating readily bonds with the aluminium. After dipping, the treatment is very similar to that employed in galvanizing.

In a more recently developed process[13] the sheets enter the molten aluminium from a sodium vapour and nitrogen atmosphere. The sodium improves the quality the coating by eliminating deposits on the surface of the aluminium.

Other methods of hot dipping employ a fluxing treatment in a molten salt mixture to provide a clean steel surface. Dipping for a few seconds in a flux pot, maintained at 400°C, is followed by immersion in the aluminium bath which is covered with a film of fluxing material and which is maintained within the range 700–950°C. Gittings[14] used a one minute, molten zinc ammonium chloride flux at 400°C. A mixture of NaCl, KCl, AlF_3 and $NaAlF_4$ at 700°C has been used[15]. A variety of fluxes such as molten halide flux mixture, solutions of halide salt/salts in water and linseed oil have been tried by Arora[16] for aluminising steel and strip.

Fluxless methods of hot-dipping are possible. The steel can be cleaned, pickled electroplated with tin, dried and then dipped. The Aluminium will dissolve the tin and wet the steel.

Aluminizing equipment is similar to galvanizing equipment with the possible exception of the aluminium holding furnace. Molten aluminium dissolves metals hence refractory lined furnaces are required in lieu of the metal pots frequently used in galvanizing.

Inasmuch as experimental data have shown that the alloy layer of commercially pure hot-dipped aluminium coatings on steel is composed of iron-aluminium constituents containing about 45% iron. The inner layer is of iron-aluminium intermetallic compound[17]. The compound layer is primarily Fe-Al with lesser amount of Fe_2Al_5[18]. The compound is brittle and should not exceed 0.01 mm in thickness if the coating is to possess satisfactory mechanical properties[19].

Experiments have shown that the thickness of the alloy layer is decreased more than 50% by the addition of 1.5—3.75% of silicon to the aluminium bath[14]. A typical microstructure[18,20] of a Si-Al coating consists of dark grey accicular needles in the outer coating layer (Si-rich).

In cases wherein a flux blanket is not used, only a thin layer of oxide is found to cover the molten metal which is the characteristics of aluminium. The oxide does not build up as in other cases. As in galvanizing, dross formation takes place but the iron content may be reduced to about 1.5% by lowering the bath temperature to about 650°C and sludging.

Pure aluminizing is rarely done. The principal alloying elements are silicon and iron. Silicon reduces the melting point of aluminium thus lowering the minimum permissible operating temperatures. Also the ductility of the coating is very much improved compared to pure aluminised coatings. However, alloys containing more than 3% silicon darken during weathering, and the best silicon content is 2 to 3%. Other addition agents such as beryllium, sodium, boron, titanium, chromium and molybdenum are added in small amounts to obtain bright, lustrous and complete coatings on steel[14].

Applications: The resistance of steel both to atmospheric corrosion and to oxidation on heating is improved by coating the steel with aluminium, and aluminium coated steel is used for roofing, siding, building, hardware, fasteners, automotive exhaust mufflers, and wire and pole line hardware to support and fasten aluminium hardware. It finds a variety of applications in the automobile, heating and building industries [18,21].

TINNING :

Hot dip tinning of iron and steel has been practised for over 200 years, first to give an attractive coating which prevented rusting, and later to protect iron equipment and steel containers used in food processing and food storage. The corrosion resistance of tin is good. Indoors it remains bright. Outdoors a protective but dull grey oxide film may form on the surface. The corrosion rate is low in rural areas and higher in marine and industrial atmospheres. The corrosion rate is about half of the average rates for zinc and cadmium, but tin is not sacrificial and must be pore free to provide complete protection. Dipped coatings are in the range of 0.15 to 0.70 mil thick.

After the usual cleaning procedures, fluxing the steel surfaces may be done in one of the three different ways: (a) as an aqueous solution in which the work is dipped before immersion in the molten tin; (b) as a molten layer blanketing the top of the tin bath; (c) as a solution or paste, or as an admixture to tin powder,

that is applied to the surface of the work prior to wipe tinning. The compositions of aqueous flux and the flux that covers the molten tin are given in Metals Hand-Book[22].

The article or the sheet wet with flux solution is lowered gently into the flux-covered tin. It is essential that fluxed objects be tinned immediately without undue delay, because the flux tends to oxidise on the iron surface, and delay between fluxing and tinning increases the possibility of defective coatings. An immersion rate of about one inch per second is generally slow enough to prevent spitting and fast enough to avoid drying.

There are three methods used for continuous pot tinning, depending on the required quality of the resulting coat. Single-pot tinning is used to obtain a coating preliminary to soldering or to parts not requiring the highest quality of finish. The single immersion pot operates with a molten flux cover at a temperature of 280° to 325°C. In the two-pot method of tinning, the work is first dipped in a tin bath with a flux cover and immersed in a second tin pot covered with oil or molten grease. This process is used to produce a high quality, and offers several advantages over single-pot tinning. The second pot can operate at a lower temperature 245° to 250°C. In cases where the articles are recessed, or from which drainage of excess metal is difficult, or with light gauge work where naturally rapid cooling would produce too heavy a coat, three-pot tinning is used; after the two pot tinning as described above, the work is immersed in a bath of hot tallow or palm oil. This promotes drainage of excess metal and the formation of a uniform coating of economical thickness, free of droplets.

A coating of hot-dipped tin consists of an inner layer of rectangular cyrstallites of $FeSn_2$[23] containing 80.0% tin, which may from rapidly at the steel-tin interface at 150°C and are stable up to 496°C. The inner layer of intermetallic compound limits the formation of additional $FeSn_2$ by restricting the diffusion of the reactants through it. It is for this reason that the compound layer in tin plate does not attain the thickness obtained in galvanized coatings. The compound layer in the tin plates is of the order of 0.5μ. The properties of the intermetallic layer and its possible role in the corrosion of tin-coated steel has been studied by Covert et al[24].

Dross: The oxidation of tin is found to be negligible and also it is kept to a minimum because of the flux blanket that is used invariably in hot tinning. The reaction between tin and iron is slow at usual tinning temperatures and so contamination of the bath because of dipping the objects in molten metal is neglible. There is some tin-iron compound formation from interaction between the tinning pot and the tin. This is also very small if the heating of the pot is properly designed. The most important sourse of contamination is the reaction of the zinc chloride flux with the iron or steel being tinned. The zinc chloride flux containing water is partially hydrolysed to form hydrochloric acid which attacks the iron, thus effecting the desired cleaning of its surface, but also forming ferrous chloride. This reacts with molten tin to give stannous chloride and free iron which dissolves in the tin to give the compound $FeSn_2$. This contamination by iron cannot be prevented since the flux reactions are an integral part of the tinning process but the rate of contamination can be kept to a minimum by proper precautions. The iron–tin compound is slightly more dense than molten tin and tends to sink to the bottom of the pot. Many methods and proprietary cleaning compounds are available for the removal of the dross.

Addition agent : Invariably tinning is done with pure tin and no other addition agents are added to the bath. A special class of coating wherein tin is alloyed with lead, commonly known as terne coating is discussed at a later stage.

Applications : Tin, being non-toxic, is the ideal protective coating on steel and iron in contact with food and dairy products. The most important application is in food handling equipment and domestic hardware, examples being pans, steamers etc. The canning industry presently is the largest consumer of hot-dip and electrotinned steel The principal engineering users of tin plate are the automobile industry, the electrical and electronics industries, the gas industry, and the photographic industry.

Lead and Terne Coating :

Pure lead is seldom used for coating purposes because of the poor adhesion with the basis metal. Pure lead coatings show a rather pronounced tendency toward formation of pinhole perforations as the coating solidifies. In the case of sheets such perforations can be removed by rolling the sheet after it has been coated On articles of irregular shape, however, this cannot be done.

In order to form a bond for a lead coating, it has been the practice in the past to make use of some alloying element common to two metals, lead and iron. Tin has been used for this purpose for many years as it alloys readily with both metals. As little as 1% tin will promote a hot dip bond, but the bonding layer will form more readily with higher percentages of tin.

By alloying lead and tin a coating is selected to favour specific advantages: high tin for a bright white colour, high lead for low cost, eutectic alloy for low dipping temperatures, solder compositions for rapid wetting and soldering, and terne alloy for moderate cost and formability.

The method: After the usual cleaning treatment, the object is fluxed and introduced into the molten metal/alloy bath. In certain cases wherein the alloy consists of less than 20% tin, no fused flux cover is used.

Nature of Coating: Unlike other metallic coatings, one does not observe any interfacial layer between steel and the lead coatings[25]. It is established that in the case of pure lead coating, only mechanical bonding exists between the coating and the basis metal.

With the increasing addition of tin, the formation of intermetallic compound[26] $FeSn_2$ is facilitated which, in turn, helps in bonding with steel. With small percentages of tin it takes a longer time or a higher temperature to form a stable intermetallic compound.

Excellent lead-coated sheets can be got by using galvanised or tinned sheets. Bray's[27] investigation of lead-dipped coatings applied over zinc coated steel reveals that a smooth, adherent continuous coating of lead may be obtained within narrow limits of bath temperature, provided the molten lead contains fractional percentage of zinc. The binding agent appears to be needle-like crystals of zinc which extend into the lead layer and attach themselves through a solid solution of zinc in lead.

Dross: Oxidation and drossing losses are less than that of other hot dip coatings. The life expectancy of the dipping pot is also greater because of the lower bath temperature required and the lower solubility of of iron in lead.

Addition agents: The major alloying element used is tin in varying proportions. Addition of more than 99.5% and up to 80% tin to lead caused the highly reflective, white appearance of a pure tin coating to be marred by an increasing spangled appearance, similar to that seen on hot-dipped galvanized steel. Coatings containing between 30 and 70% of lead had a reasonably reflective coating. The coatings containg only 10 or 20% tin present a dull grey featureless surface with very little reflectivity. Thwaites[28] studied the tin-lead alloy coating in the composition range 0.5% to 90% lead.

Applications: Lead-coated sheet may be very severely deformed without having the coating stripped off; hence, such material finds considerable application in deep stamping operations in which the coating aids materially by acting as a lubricant. Lead is the only common coating metal which is resistant to sulphuric and sulphurous acid fumes.

Tin-lead alloy coatings find uses where nontoxicity is not essentil and the cheaper coating can safely be used. Examples are radiator or heat exchanger units, petrol tanks and filler pipes and in the electrical field for chassis, relay cans and tags where solderability is particular, and also protection against corrosion is the chief concern.

Alnminium-Zinc Alloy Coating

Until recently only single metal such as zinc, aluminium, tin and lead was used for producing protective coating by hot dipping over steel and other materials.

Recently investigators[29-32] have come out with a new alloy coating. namely, Al-Zn alloy coating which combines the best properties of both galvanizing and aluminising and hence superior to both of them. So far the coating has been accomplished by two methods: (a) roll bonding[29], and (b) hot dip process[30]. A simpler method[33] of coating the alloy over steel is by using an aqueous flux mainly based on ammonium chloride. Coatings having 30 to 50% zinc have been obtained.

Method: After the usual procleaning. the objects are dippeoud in an aqueous flux, dried and immersed immediately in the molten alloy for a few seconds. withdrawn and air cooled.

Nature of coating: The coating consisted of three layers, the base steel followed by an intermediate layer and the outer coating of Al-Zn alloy layer. The

intermediate layer is found to be thin and 3-5 times thinner than the alloy coating.

Dross: Due to the reaction of the molten metal with the flux and iron and dross is produced, which settles at the bottom. Since the temperature of operation is high compared to galvanizing, ash is formed which floats over the molen metal.

Applications: The new alloy coating of aluminium and zinc combines the best properties of both. It has the durability in marine and industrial atmospheres, and the high temperature oxidation resistance and heat reflectivity characteristic of aluminium coatings, with the formability and galvanic protection of cut edges characteristic of galvanized coating. In U. S. A. the alloy coated materials are being tested for use in silo roofs, grain bins and similar agricultul applications. Other potential uses include gutters, downspouts, duct work, residential siding, utility sheds, heating and air conditioning components, and formed metal shingles.

It is appropriate to mention here that the corrosion tests have indicated that the corrosion-resistance of this alloy coating is much superior to both galvanized and aluminized coating. The coating protects the base steel by sacrificial action.

Post-treatment: If the coated parts are held at elevated temperature, growth of the alloy layer can continue even though the coating has solidified. The coatings are to be cooled immediately by quenching, blast cooling or air cooling. Unwanted alloy growth can damage adherence of the coating and cause bright coatings to become dull and unattractive.

Finishing operations for hot dipping include the removal of flux residues, hard residues formed due to dross etc. In certain cases, the articles may by stained (eg. yellow oxide stain in tinning) which can be removed by momentary dip in cold 1% hydrochloric acid. After the acid dip they are given a neutralizing rinse in dilute alkaline solution followed by rinsing in clean water.

Conclusion :

The characteristics of all the hot-dip protective coatings on steel are given in Table 2. From the existing technology, the following may be said. Zinc coating technology is well known and the subject has been studied intensively. Even though the use of galvanized steel is increasing at present, little change in the standard practice can be expected in the future.

Although aluminium coatings have been in use for many years, the mass production of coated sheets are still under development. A standard procedure may be expected to be established in the near future. It is reasonable to expect that the demand for aluminium coatings will increase and may replace at least partially the zinc coatings on many applications.

Because the world wide eonsumption of tin has out paced its production and consequently raised its price tin plate producers are attempting to replace wherever possible by other metals such as terne coating.

The use of lead coatings and terne plate can be expected to decline because of the severe competition such as plastic coated steel, PVC clad steel etc.

Table—II Characteristics of hot-dip coatings on Steel

Characteristics	Zinc	Aluminium	Tin	Lead	Terne	Al-Zn alloy
Colour	White	White	Bright	dull	bright to dull	White
Corrosion resistance	Fair	Good	Good	Good	Good	Good
Corrosion product	Flaky	Flaky	Tight	Tight	Tight	Flaky
Sacrificial protection	Yes	Yes	No	No	No	Yes
Alloy with steel	Yes	Yes	Yes	No	Yes	Yes
Alloy layer	moderate	moderate	thin	None	thin	moderate
Melting temp. °C	419	660	232	327	varies	varies

Developments in composite coatings are of interest at present. Coating such as zinc-aluminium has been developed with better corrosion resistance in many applications than the conventional coatings.

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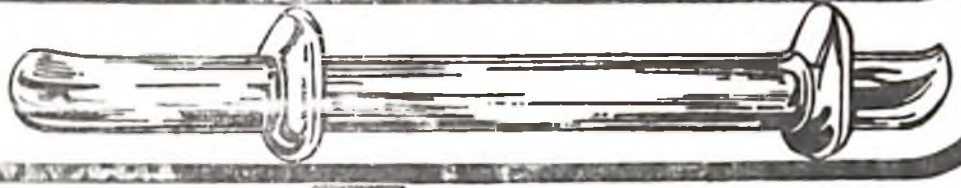
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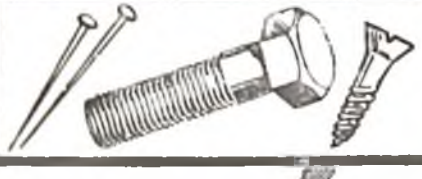
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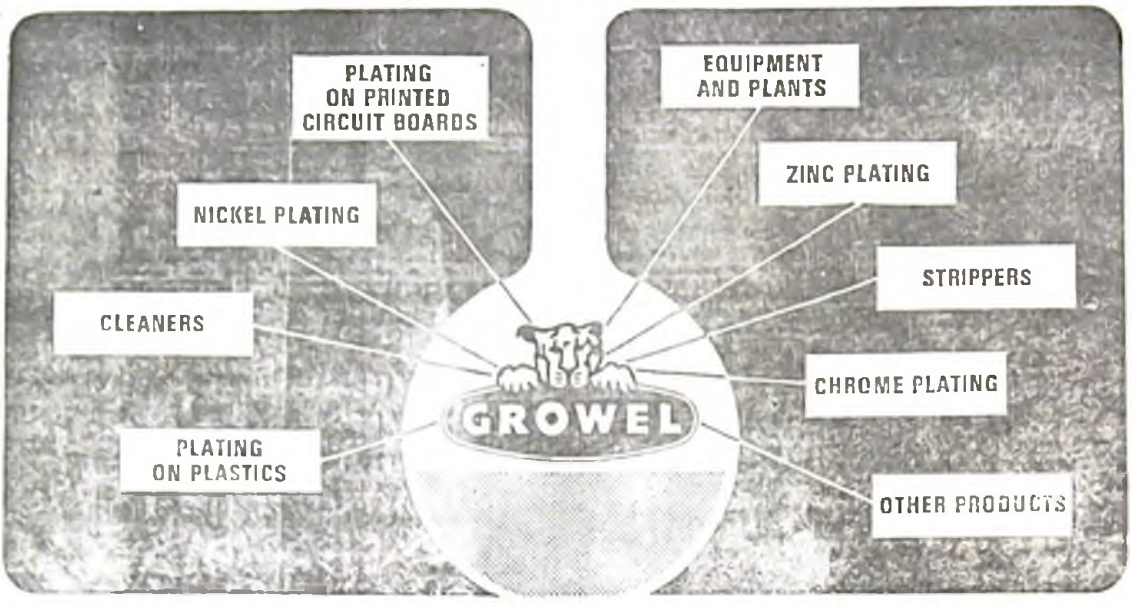
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