

Conservation of non-ferrous metals by protective treatments and substitution by aluminium

Defence Research Laboratory (Materials), Kanpur

ALTHOUGH non-ferrous metals corrode at a lesser extent than steel, they suffer appreciable attack on exposure to atmosphere and when used in underground service.

The corrosivity of non-ferrous metals have been determined by DRL(M), Kanpur, by outdoor exposure at Bombay, Cochin, Balasore and Jodhpur. Corrosion of non-ferrous metals buried in Kanpur soil has also been determined. The corrosion rates are shown in Tables I and II.

TABLE I Atmospheric corrosion rate of non-ferrous metals at various sites

Metals	Corrosion rates (mgm./sq. dm/year)			
	Bombay	Cochin	Balasore	Jodhpur
Copper	87	82	338	32.5
Brass	69	60	69	8.8
G. I. (coating thickness=0.0014 inch)	293	186	432	27.3
Lead	30	46	—	—
Aluminium	—	9	48	0
Zinc	242	129	1977	124
Iron	3266	5104	5260	336

Passivation of non-ferrous metals

Conservation of non-ferrous metals can be achieved by prolonging their life by application of various passivation treatments. The following treatments have been applied by DRL(M) on a number of components made

TABLE II Underground corrosion rate of non-ferrous metals at Kanpur after one year exposure

Metals	Corrosion (loss in gm./sq. ft.)
G. I.	8.4
Aluminium	1.62 2.1
Lead	26.7
Iron (cold drawn sheet)	84.45

of non-ferrous metals and alloys and satisfactory results have been observed.

Tin

Tin cans have been given a 30 seconds dip at 85°C in the following solution. Corrosion of treated cans by card-board cartons used for packaging was prevented.

Sodium dichromate	—	3 gm
Sodium hydroxide	—	10 gm
Wetting agent	—	2 gm
Water	—	1 litre

The above treatment was also found to be beneficial for tin-plated steel components.

Zinc

Zinc-coated articles were immersed for 5 to 10 seconds at room temperature in a solution containing 200 gm of sodium dichromate crystals and 6.6 ml of concentrated sulphuric acid per litre of water. Corrosion tests carried out on such passivated surfaces (Aircraft

process specification No. DTD 923A) have shown improved corrosion resistance.

In marine atmosphere, galvanised iron roofings, which corroded severely in a few months, were treated by brushing with lithoform (ICI proprietary product). Lithoform treated surfaces subsequently painted with one coat of red oxide zinc chromate primer and two coats of G. S. brushing aluminium showed excellent corrosion resistance for more than 2 years.

Aluminium

Aluminium surfaces can be protected against corrosion by the modified Bauer Vogel treatment. The process consists of an initial cleaning with 10% nitric acid (3 min.), washing in water and dipping for 5 minutes in a solution (at 90°C) containing 5% sodium carbonate, 1.5% potassium chromate in water. The article should be agitated during treatment and afterwards washed with water and dipped for 15 minutes at 90°C in a 5% sodium silicate solution. It is then rinsed in water and dried.

For articles which cannot be suitably dipped and treated as above, a paste of the following composition should be prepared, applied by brush and washed away after 15 to 30 minutes:

Potassium chromate	—	8 lb
Anhydrous sodium carbonate	—	3 lb
Caustic soda	—	3 lb
Water	—	1 gallon.

Aircraft components have been treated with the paste composition given above to increase corrosion resistance.

Brass

Dezincification is a type of corrosion peculiar to brass resulting in the loss of zinc and a copper-rich residue is left.

A drop-test to determine the susceptibility of brass to dezincification has been developed, which consists of application of a drop of 20% cupric chloride solution in water on a freshly cleaned surface of brass. Development of red colour in the drop within 30 seconds indicates susceptibility to dezincification.

The two following passivation treatments of brass to prevent dezincification on exposure to salt fog have been tried i.e. DRL(M) with success:

- Degrease in trichlorethylene, pickle in 50:50 hydrochloric acid solution for 3 minutes and rinse in water. Treat for 10 minutes in a solution at 30°C containing 60 gm of chromium trioxide, 44 gm of concentrated sulphuric acid (24.4 ml) and 500 ml water. Rinse in cold water and then in hot water. Dry at 100°C.
- Pickle in a solution containing 100 gm chromium trioxide, 60 ml of concentrated sulphuric acid and 1 litre of water. Wash and passivate in a solution containing potassium chromate (50 gm),

sodium chloride (2.5 gm), sodium sulphate (12.5 gms), concentrated sulphuric acid (1.5 ml) in water (500 ml) for 10 minutes at room temperature. Rinse in water and dry at 100°C.

Both the above passivation processes have been found to be effective, the former being more effective than the latter. Brass cartridge cases susceptible to dezincification (as shown by 30 seconds spot test) have shown considerable resistance (4 hours in spot test) after passivation treatment.

Copper

Attempts are being made to ascertain the possibility of substituting copper with aluminium for earthing plates.

Better design to prevent corrosion

Proper designing of assembled metallic articles can also prevent failure in service. Dissimilar metals which promote galvanic corrosion should be avoided. If this cannot be avoided, the metals should be electrically insulated from each other. Cases are known where copper dissolved in water was plated out on baser metal like iron and aluminium, causing galvanic corrosion. Stray current from electric tram and rail tracks have caused corrosion failure of underground structures. Crevices cause stagnation leading to corrosion. Flue gas exits should be designed so as not to allow 'dewing' of acidic residues which encourages corrosion. Where necessary, proper aeration should be provided to avoid formation of differential oxygen cells. Local concentration of electrolytes should be avoided. Box sections, crevices, channels should be avoided. Provision should be made for easy and complete drainage and cleaning of electrolytes from equipment. Contact with corrosive non-metallic materials or gases liberated therefrom, should be prevented.

Use of aluminium

Conservation of scarce non-ferrous metals can be affected by substituting them with aluminium, which is indigenously available. Aluminium has been successfully used as electrical conductors in place of copper¹ as sheath for electrical cables in place of lead² and as a structural metal in general engineering field in place of steel. It has become a common practice today to replace nickel and chromium-plated parts with bright anodised aluminium.³ Investigations were undertaken in Defence Research Laboratory, Kanpur, to effectively replace some copper-based alloys, white metals and precious metals like silver, gold with suitably treated aluminium. These include brass or white metal button, badges, buckles, metal components of web equipment, suspension chains, instruction plates, quick release boxes for parachute, electro-plated wares requiring nickel and chromium platings. For use on ceremonial occasions, gold and silver platings on brass badges, buttons and insignia are also required. Silver and chromium plated reflectors, telescopic type aerials, binocular tubes, etc. are also

made of non-ferrous metals. These have been successfully replaced by properly finished aluminium.

Anodising

In most cases, aluminium of various grades are anodised to increase the corrosion and abrasion resistant properties. Anodic treatment in acidic solution is carried out to produce a thick oxide coating. The anodic coating is micro-porous and, when freshly prepared, can absorb dyes, pigments, lacquers, etc. As such, it is possible to impart to it various metallic shades by dyeing to match various camouflage colours.

Anodising process for both super purity (99.99%) and commercial purity aluminium were standardised. To produce high reflectance as is required for reflectors, mirrors, motor car trim or for gold and silver shades, high purity aluminium was mechanically polished and then electrolytically brightened making the article as anode by the following process :

Electrolyte	{	Sodium carbonate	15%
		Trisodium phosphate	7%
	Temperature	75°-80°C	
	Cathode	Steel	
	Current density	50 amp/sq. ft.	
	Time	About 15 minutes	

This removed fine scratches from the surface and reflectivity above 95% was obtained. The next process consisted essentially of anodic treatment in 8% w/v sulphuric acid solution at 8 amp/sq. ft. current density at 25° ± 0.5°C. The conditions of anodising were so controlled that the rate of formation of anodic oxide only just exceeded the simultaneous rate of solution of the oxide film. By this means a uniformly bright surface of high reflectivity and transparency was obtained.⁴ For increasing the corrosion resistance of the film, final sealing of the oxide coating was carried out in steam. The shade produced resembled in appearance with those of silver, chromium and white metal. For producing golden appearance, the anodic coating was dyed in a mixture of red and yellow dyes before the sealing treatment. As super purity aluminium is imported, experiments were also carried out to standardise a process for producing bright anodised surface by using indigenously produced Indal aluminium of the highest available purity of 99.7%. The reflectivities of bright anodised surface with other metals have been compared and shown in Table III.

For commercial grade aluminium, a good general purpose electrolyte was found to be one containing 14% w/v sulphuric acid operated at 20°-25°C at 12 amp/sq. ft. current density. This gave hard and abrasion resistant coating and by dyeing; brass, bronze, black, olive green or any other shades could be easily produced.

The properties of anodic coating is largely dependent on the processing conditions and alloys used. It has a diamond pyramid hardness between 300 and 700 VPN and diamond scratch hardness greater than that of high

TABLE III Reflectivity of anodised aluminium compared to other metals

Specimen	Total (per cent)	Specular (parts in 100)	Diffuse (parts in 100)
Super purity aluminium (99.9%) (electropolished and anodised)	84.1	99.4	0.6
INDAL Aluminium 99.7% (electropolished and anodised)	55.0	—	—
Stainless steel	59.5	97.0	3.0
Chromium plating	63.0	99.7	0.3
Rhodium on nickel plating	69.1	99.5	0.5
Lacquered silver plating	89.8	96.5	3.5
20% nickel-silver alloy (cast)	64.3	—	—

speed steel (900-VPN). The coating has excellent corrosion results obtained at DRL(M), Kanpur, of abrasion resistance and corrosion resistance of various thicknesses of anodic film as produced on superpurity aluminium and various grades of Indal aluminium are shown in Tables IV and V. Abrasion test was carried out on

TABLE IV Abrasion resistance of anodic coating on different grades of aluminium

Description of aluminium	Thickness of anodic coating (mm)	No. of cycles of failure
Super purity aluminium	0.003	850
" " "	0.007	3000
" " "	0.0127	7000
" " "	0.0177	7300
INDAL 99.7%	0.0037	700
" " "	0.0078	2300
" " "	0.0130	6800
" " "	0.0184	6800
INDAL 99.5%	0.0035	700
" " "	0.0076	2500
" " "	0.0127	6600
" " "	0.0177	7500

the Martindale-Abrasion testing apparatus under a load of 1 lb/sq. inch against '0000' grade emery paper. Corrosion test was carried out by spraying the specimens with synthetic sea water once every twenty-four hours

TABLE V Results of corrosion test

Description of panels	Thickness of anodic coating (mm)	Gain in wt. (mgm/cm ²) due to corrosion	Condition of surface after corrosion test
Super purity aluminium	Not anodised ; electropolished only	10.76	General staining only no bulky white corrosion
" "	0.003	2.54	No visible change
" "	0.0127	1.76	" "
" "	0.0177	0.98	" "
INDAL 99.7%	Not anodised ; electropolished only	11.54	Bulky white corrosion spots at places
" "	0.005	2.32	Few white spots ; no bulky white corrosion product
" "	0.0152	1.74	" "
" "	0.0254	2.13	" "
INDAL 99.5%	Not anodised ; electropolished only	10.76	Few bulky white corrosion spots
" "	0.0035	2.73	Few white spots at places
" "	0.0127	1.56	" "
" "	0.0177	1.37	" "

and then keeping them suspended over a dish of water to maintain 96–100% relative humidity at a temperature between 30° and 33°C. The corrosion test was continued for a duration of 30 days.

Paint/primers

The conservation of copper, lead and zinc powder/pigments in paint formulations is possible by the use of aluminium pigments (both leafing and non-leafing type), which are manufactured indigenously in the country.

Flake aluminium powder and aluminium paste for paint use are made by wet ball milling of the granular form of aluminium. For ball milling, the fine particle size granulated aluminium is placed in a cylindrical mill together with steel balls, lubricant (stearic acid) and a dispersing liquid (mineral spirit). The final product obtained after milling is leafing type aluminium pigment in paste form. The coating of aluminium stearate formed during milling operation makes possible the leafing action of the flakes in a paint film with the resulting brilliant metal finish. Flake aluminium powders are produced by complete drying of the paste through evaporation of the mineral spirit under vacuum. These may be treated to convert them into the non-leafing type or a special lubricant may be used which produces the non-leafing type directly.

Lead and zinc compounds in wide use in paint industry are : lead-chrome, white lead, basic sulphate of lead,

lead cyanamide, calcium plumbate, basic lead silico-chromate, red lead, zinc oxide, zinc chrome, zinc tetroxochromate, etc. Out of these white lead, zinc oxide, lead-chrome find their use as prime pigments in finishing paints. Aluminium pigment, because of its characteristic silvery bright metallic appearance, cannot replace/substitute these pigments in finishing paints.

Lead and zinc pigments like red lead, basic lead sulphate, lead cyanamide, basic lead silico-chromate, calcium plumbate are used as rust inhibiting pigments. Zinc oxide cannot be considered a corrosion inhibitive pigment but it is frequently used with other pigments in metal primers because it is highly reactive with acidic materials and thus reduces corrosive conditions. It tends to neutralise acids absorbed from industrial atmospheres also. Lead and zinc metallic powders are used in formulation of metallic lead and zinc rich primers. It is now intended to find out how best, the two types of aluminium pigments can substitute/replace the above mentioned rust inhibiting pigments/powders in priming paints. These can fulfil the requirement only if they possess rust inhibitive property and suitable primers can be formulated by using aluminium pigments as such or in combination with other pigments.

Each aluminium particle is coated with an extremely thin and impermeable oxide layer as well as a layer of the fatty acid used as lubricant in the manufacturing process. The aluminium particles are thus passified and so have a considerable resistance to corrosion currents and can be considered to be electrically inactive pigments. This electrical resistance coupled with the chemical passivity and the comparative impermeability caused by their orientation in a paint film all add to the usefulness of aluminium powder as anti-corrosive pigment. Aluminium pigment is a very popular constituent of paints for protection of steel work against corrosion, not only in the undercoat and finishing coats but in the primer also. In other words protective painting systems may be based entirely on aluminium paints where aluminium paste pigment is used in primer. There is no advantage in increasing the content above about two pounds per gallon because this encourages brush marks to be left and corrosion may start at the brush marks, where the paint coating may be exceptionally thin. Aluminium primers give good performances under salt water or marine conditions.

From the above it will be noted that aluminium powder shows its usefulness as an anti-corrosive pigment. Leafing type aluminium pigment does not find its use in priming paints, as it imparts a smooth and lustrous finish to the priming coat, which will impair the adhesion of subsequent coats. Only non-leafing type aluminium powder can be used as such or in combination with other pigments in priming paints. If such formulated primers can give satisfactory corrosion resistance property, like those of red lead and zinc chrome primers, their use in lieu will very much help in relieving pressure on the use of primers based on lead and zinc powder/pigments manufactured from imported lead and zinc.

Work⁵ carried out by Defence Research Laboratory (Materials), Kanpur, has confirmed that non-leafing aluminium pigments in combination with zinc-oxide in

TABLE VI Rating classification of primer

	Good (10-8)	Fair (7-5)	Bad (4-0)
(1) Humidity cabinet test (10 days) (IS : 101-1961) One coat of primers 1 to 5 and one coat of finishing	1, 2, 3 & 5	4	—
(2) Salt spray test (10 days) (IS : 104-1962) One coat of primers 1 to 5 and one coat of finishing	1, 2, 3	4 and 5	—
(3) Outdoor exposure (16 months) One coat of primers 1 to 5 and one coat of finishing	1, 2, 3, 4 & 5	—	—

Note : (i) 10 denotes no corrosion and zero (0) denotes cent per cent corrosion.

(ii) Primers 1 to 5 were the same as given in Table VIII.

a priming paint with a top coat show comparable corrosion resistance property to the conventional red lead primer. Results are given in Table VI. Outdoor exposure carried out under semi-industrial climate conditions of Kanpur for four years also exhibited similar performance (Table VII-findings confirmed by Regional Research Laboratory, Hyderabad). This paint, however, without a top coat exhibits inferior corrosion resistance property to red lead primer both in laboratory corrosion tests and out-door exposure. Results are given in Tables VII and VIII. On the basis of above work IS specification IS : 2931-1964 for Ready Mixed Paint, Brushing, Aluminium Zinc Oxide Composite Primer has been drawn.

TABLE VII Results of outdoor exposure

Primer No. and mode of application	Primer (one coat)			Primer with a finishing coat	
	6 months	6 months	6 months	16 months	38 months
1. Brushing	10	10	10	10	9
2. „	10	10	10	10	—
3. „	8	3	10	10	—
4. „	9.5	7.5	10	9.5	8
5. „	2	1	10	10	10
6. Spraying	10	10	10	10	—

Aluminium Zinc Oxide Primer possesses several advantages over red lead primer, and it can be manufactured from indigenously available ingredients. Work has, therefore, already been started by Defence Research Laboratory (Materials), Kanpur, to study its protective performance vis-a-vis lead primer by carrying out out-door exposure at several stations in the country representing varying climatic conditions (all other conventional primers are also included in the trial) and to find out how far it compares so that it can be recommended to replace red lead primer which involves considerable foreign exchange.

Aluminium Zinc Oxide Primer has also shown very encouraging results as a primer⁶ for aluminium surfaces and this can very suitably replace zinc chrome primer so far considered the best for aluminium surfaces.

After long use of 'Admar' Anti Corrosive Primer for ships, it was felt that an improved formulation was needed based on a more water resistant medium and less liable to blistering. Investigations⁷ showed that this could be achieved by incorporating some tung oil in medium and aluminium powder in place of white lead in the pigment. The modified formulation with better performance has been adopted for use on all new vessels.

Recent work⁸ carried out in foreign countries has shown that leafing type aluminium pigment can partly replace leafing zinc dust in formulation of zinc rich primers with lower pigmentation.

It has been reported^{9,10} that suitable primers for iron and steel have been developed by use of aluminium pigment in combination with red mud, barium potassium chromate, manganese ore/manganese dioxide, which are all indigenously available. Red mud is a waste by-product of aluminium industry available in abundance (several thousand tons) in our country. Regional Research Laboratory, Hyderabad, has established the technical know-how of the manufacture of barium potassium chromate from indigenously available raw materials. Manganese ore and manganese dioxide are also indigenously available. Possibility may, therefore, be explored to develop primers on the basis of above formulations and find out how far they can find their use in replacing conventional primers based on lead and zinc powder pigments. Work on these lines has already been started at Defence Research Laboratory (Materials), Kanpur.

From the above it will be seen that aluminium pigment as such and in combination with other pigments can be utilised in developing suitable primers for iron and steel thereby replacing conventional primers based on lead and zinc powder/pigments involving considerable foreign exchange.

Sprayed aluminium coatings

The importance of high temperature oxidation study of metals is evident from the fact that in the following processes metals, ferrous and non-ferrous, are exposed to high temperature : carburising, cyanide hardening, exhaust systems, exhaust manifolds, preheating and recuperating, superheating, enamelling, annealing.

The oxidation of sprayed coatings (thickness $\sim 0.005''$,

TABLE VIII Results of laboratory corrosion tests

Primer description	Mode of application	Humidity test IS : 101-1961			Salt spray test IS : 104-1962			A.R.E. Salt drop-let test BS : 1391-1952		C.R.L. Beaker sulphurdioxide test BS : 1391-1952		Average overall tests
		4 days	10 days	20 days	4 days	10 days	20 days	4 days	10 days	4 days	10 days	
1. Red Lead Primer, IS : 102-1962.	Brushing	10	10	9	10	9	9	10	9	10	7	9.3
2. Red Lead/Red Oxide Primer Def. Specn. IND/SL/3135 (a)	do	10	10	9	10	9	9	10	9	9	6	9.1
3. Zinc Chrome Primer IS : 104-1962.	do	9	8	8	9	8	8	9	9	7	3	7.8
4. Red Oxide/Zinc Chrome Primer IS : 2074-62.	do	6	5	3	7	3	2	8	2	2	1	3.9
5. Aluminium Zinc Oxide Composite Primer IS : 2074-62	do	8	7	5	1	1	1	2	2	4	3	3.4
6. do	Spraying	7	6	4	3	3	2	4	3	5	5	4.2
7. Zinc Rich Primer	Brushing	8	8	4	4	3	3	6	4	10	10	6.0
8. Metallic Lead Primer	do	10	10	9	9	9	8	9	9	10	9	9.2

Note:—(1) Tests were carried out on mild steel panels painted with one coat of priming paint as such (no top coat was used).
(2) 10 denotes no corrosion, zero (0) denotes cent per cent corrosion.

0.007" and 0.008") of aluminium on mild steel has been studied in DRL (M) at various temperatures between 700° and 950°C. Initially 1"×1" mild steel panels were taken and the following treatments were made.

- No treatment—(control).
- Aluminium sprayed (0.005" and 0.007" thickness).
- Aluminium sprayed (0.005" and 0.008" thickness), coated with bitumastic paint and heated to 800°C for 15 minutes.
- Aluminium sprayed (0.005" and 0.007" thickness) treated by an immersion for 30 seconds in ethyl silicate dope, allowed to drain and age for 48 hours at room temperature. The dope was prepared¹¹ by adding 81 parts (by vol.) of alcohol to 30 parts of 0.06% hydrochloric acid solution followed by 190 parts of ethyl silicate. The well shaken mixture was allowed to stand for 1 hour and 151 parts of ethyl silicate was further added.

The oxidation test was carried out by heating the above mentioned panels for 3 hours at each temperature (700°, 750°, 800°, 850°, 900°, and 950°) and determining

the gain in weight at each stage. The data are given in Table IX.

Discussion

Heat treatment produces three distinct layers.^{12,13} Next to the basis steel, there is a layer of solid solution of iron and aluminium, then an alloy layer of iron and aluminium rich in aluminium. Finally there is a thin coating of Al₂O₃. The solid solution layer of iron and aluminium is highly heat-resistant. Comparing the gain in weight for .005" thick sprayed aluminium coated and heat-treated panel with .008" thick coated and heat-treated panel it can be seen that the gain in weight by oxidation is much less in case of .008" thick sprayed coating over the whole range of temperature studied. From 800°C and above the successive gain in weight in case of .008" thickness is also much less than that of .005" thickness at corresponding temperatures.

The heat resisting property of sprayed aluminium panel which has been doped with partially hydrolysed ethyl silicate is due to the deposition of silica¹¹ in the pores of sprayed metal. Comparing the data for .005" and .007" thick sprayed coatings which have been doped

TABLE IX Weight gain in mgm/sq.in./3-hours of aluminium sprayed panels

Temperature °C	Heat treated (800°C) coating thickness		Ethyl silicate doped (not heat treated) coating thickness		Not heat treated coating thickness		Mild steel panel
	.005"	.008"	.007"	.005"	.005"	.007"	
700	2.88	2.26	—	11.09	22.58	4.40	11.30
750	6.72	5.41	2.36	17.48	28.79	15.38	24.50
800	15.95	7.86	8.90	25.90	37.58	19.99	52.50
850	37.45	14.61	20.60	38.09	53.64	27.47	123.50
900	84.38	24.59	33.20	53.15	74.88	40.54	230.20
950	128.39	42.98	62.07	83.84	101.95	54.38	368.0*

*Extrapolated

with ethyl silicate, it is seen that gain in weight in case of .007" thickness is much less as compared to that of .005" thickness over the whole range of temperature.

Similarly the data for .005" and .007" thick coatings which have not been heat-treated indicate that .007" thick coating gives better heat resisting property. Data for mild steel can be compared with any thickness of coating or any surface treatment described here and the only conclusion is that sprayed coatings of aluminium on mild steel gives better performance than on bare steel, the sprayed coatings within themselves give better performance as the thickness of sprayed coating increases.

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