Different Sealing Methods For Anodized Aluminium & New Sealing Techniques & Fluorpolymers Sealing; Environmental Impact Recapitulation & Parameters Optimization¾ Part III

Prof. Xavier Albert Ventura, Laboratory of Electrochemical Research & Development, Integral Centre, Barcelona, Spain

This work compares different sealings explained in the papers presented previously at SUR/FIN® 2000 (Chicago) and SUR/FIN® 2001 (Nasville). It completes the first part of a five-years project o the University of Barcelona. Two mores years remain for further research. Different cold sealings are discussed: nickel fluoride, nickel acetates, nickel sulphate, molybdenum sulphate, cobalt sulphate and cobalt and molybdenum fluorides. Their performance is verified with sealing test ISO 3210 –the minimization of weight loss in sealing. The optimisation is evaluated of anodising parameters versus sealing and vice versa, as well as micro thickness and corrosion resistance. Environ mental impact is considered that can produce metallic contents of transition elements; nickel, cobalt and molybdenum. After working with transition elements sealing, we are now evaluating alkalines and alkaline-earth sealings. This work also compares some studies and tests carried out with transition elements in nickel, cobalt and molybdenum sealing as well as fluorpolymers aluminium-manganese (Al-Mg) and aluminium-manganese-titanium (Al-Mg-Ti) alloys.

For more information, contact:
Prof. Xavier Albert Ventura
Laboratory of Electrochemical Research & Development, Integral Centre
Carabela La Niña, 22, 2º, 1ª
08017 Barcelona, Spain
Phone: (34) 933 37 03 22 / Mobile (34) 639 72 10 65
Fax: (34) 933 38 92 31
e-mail: javieralbort@menta.net

Introduction

This work together with the papers presented previously at SUR/FIN® 2000 (Chicago) and SUR/FIN® 2001 (Nasville), completes this cycle of cold sealing with cobalt and molybdenum. Also, Later, we extend the cold sealing technique to fluorpolymers and organic coatings. In our future work aluminium-manganese fluorpolymers and aluminium-manganese and titanium alloys, will be discussed.

Anodic Oxidization

Unlike the electrocoating processes in which the layer increases towards the electrode outer part, in anodic oxidization the layer growth is towards the inner part of the metal and therefore consuming it.

If we concentrate on the faradic conversion of Al to oxide, we observe that for each gram of metal consumed, 1.889 gr. of oxide is obtained.

The coating relation is defined as the oxide amount formed per gram of reacted aluminium, which experimental value -double weighting measurement- show us the efficiency of the anodic process.

Non-Porous Layer

The non-porous layers are those that do not retain dissolvent in the formed oxide layer and therefore, are compact, dielectric and thinner. The layer thickness initially depends on the applied potential and temperature. 700 V and refers

The electrolytes causing this type of breakage are aqueous dissolutions of borates, tartrates, succionates, citrates, phosphates and carbonates. The breakage potential for the non porous layers, oscillates between 500 and to thickness between 7,000 to 10,000 A.

The velocity formation in the initial layer increases lineally in relation to the current density and the coating relation value near to the theoretical value of 1.889 as for a process that has not secondary reactions.

Porous Layers

Porous layers are those that do not hold dissolvent in the formed oxide layer. They are gelatinous and therefore thick and soft. In the porous layer the coating relation has a value of 1.35 to 1.46 due to the existing competitive effect of dissolution against that of oxide formation. This seems to transform the superficial layer in small crystalline nuclei of hidratated oxide which several authors believe to be boehmite. However, most probably what happens is the formation of a defective variety of pseudoboehmite Al_2O_3 x H_2O (x between 1.8 to 2.5).

We worked also with the sealing process elements like cobalt and molybdenum.

Cobalt is element 27 in the periodic table and has an atomic weight of 95.94 and, therefore, let us considers its weight as 59 with oxidation states of valences 2 and 3 like nickel and with an electro negativity of 1.8, according to Pauling.

Molybdenum is element 42 in the periodic table and has an atomic weight of 95.94 and, therefore, let us considers its weight as 96, with oxidation states of valences 2, 3, 4, 5, and 6, and with an electro negativity of 1.8, according to Pauling.

Their sealing mechanic reactions are as follows:

SEALING MECHANICS REACTION

Sealing Mechanics Reaction for Ni

1) $x\ Al_2O_3H_2O + 4F^- + 2xH_2O \rightarrow 2\ x\ Al(OH)\ F_2 + 4OH^- + 16SO_4^{=}$

2) $Y\ NiF_2 + 2yOH^- \rightarrow yNi\ (OH)_2 + 2yF^-$

3) $z\ Al_2O_3\ H_2O + 2zH_2O \rightarrow 2z\ Al(OH)_3 + 0.16z\ SO_4^=$

By adding equations 1), 2) y 3) it is obtained:

4) $(x+z)\ Al_2O_3H_2O + yNiF_2 + (4x-2y)F^- - 0.16\ (x+z)\ SO_4^- +$
$+ 2(x+z)\ H_2O \rightarrow 2Al(OH)F_2 + yNi(OH)_2 + 2\ z\ Al\ (OH)\ 3 + (4x-2y)OH^-$

After treating with H^+ the pH increases, the reaction being:

$$10^{-6.6} - 10^{-9.5})\ x \quad \frac{N51}{150dm^2} \quad = \quad 5.9 \times 10^{-5}\ \text{gr. equivalent/dm}^2$$

The analysis of the solution indicates consume values of Ni^{++} and F^- in 10 mg/dm^2. Ni and F^- atomic weight is 59 and F^- 19 and it is obtained by

$$\frac{5{,}5 \times 10^{-5} \times 10^3}{4x-2y-0.32(x+z)} = \frac{10/59}{y}$$

$$\frac{10/19}{4x} = \frac{10/59}{y} \qquad y = 1.31$$

As $4x-2y -0.32\ (x+z) = 0$ then $z = y$ assuming $x = 1$ and replacing equation 4)

$5{,}20\ Al_2O_3 \cdot H_2O + 1{,}3\ NiF_2 + 1{,}4\ F^- + 10.4\ H_2O$ _ $Al\ (OH)_3\ F_2 +$
$+ 1.3\ Ni(OH)_2 + 8.4\ Al(OH)_3\ 1.4\ OH^-$

For the new cobalt and molybdenum sealing the X-rays diffraction and tangential values should be studied.

Using the transition elements that better form the interaction Al/O in porous layer non-porous layer and barrier layer

Nickel	$Ni^{+2}\ F^-$
Cobalt	Co^{+2}
Molybdenum	Mo^{+2}

Sealing Mechanics Reaction for Co

1) $x\ Al_2O_3H_2O + 4F^- + 2xH_2\ O \rightarrow 2\ x\ Al(OH)\ F_2 + 4OH^- + 16SO_4^=$
2) $Y\ CoF_2 + 2yOH- \rightarrow y\ Co\ (OH)_2 + 2yF^-$
3) $z\ Al_2O_3\ H_2O + 2zH_2O \rightarrow 2z\ Al(OH)_3 + 0.16z\ SO_4^=$

By adding equations 1), 2) y 3) it is obtained:

4) $(x+z)\ Al_2O_3H_2O + y\ Co_2 + (4x-2y)F^- - 0.16\ (x+z)\ SO_4^- + 2(x+z)\ H_2O \rightarrow 2Al(OH)F_2 +$
$+ yCo(OH)_2 + 2\ z\ Al\ (OH)_3 + (4x-2y)OH^-$

After treating with H^+ the pH increases, the reaction being:

$$10^{-6.0} - 10^{-9.1}) \text{ x } \frac{\text{Co54}}{150\text{dm}^2} = 5.3 \text{ x } 10^{-4} \text{ gr. equivalent/dm}^2$$

The analysis of the solution indicates consume values of Co^{++} and F^- in 10mg/dm^2. Co and F^- atomic weight is 57.93 and F^- 18,94 and it is obtained by

$$\frac{5{,}5 \text{ x } 10^{-5} \text{ x } 10^3}{4x-2y-0{,}32(x+z)} = \frac{10/58.93}{y}$$

$$\frac{0/18.94}{4x} = \frac{10/58.93}{y} \qquad y = 1.31$$

as $4x-2y -0.32 (x+z) = 0$ then $z = y$ assuming $x = 1$ and replacing equation 4)

$$5{,}20\ \mathrm{Al_2O_3{\cdot}H_2O} + 1{,}3\ \mathrm{CoF_2} + 1{,}4\ \mathrm{F^-} + 10.4\ \mathrm{H_2O}\ \_\ {}_2\mathrm{Al\,(OH)\,F_2} + 1.3\ \mathrm{Co(OH)_2} +$$
$$+\ 8.4\ \mathrm{Al(OH)_3}\ 1.4\ \mathrm{OH^-}$$

Sealing Mechanics Reaction for Mo

1) $x\ \mathrm{Al_2O_3H_2O} + 4\mathrm{F^-} + 2x\mathrm{H_2\,O} \rightarrow 2\ x\ \mathrm{Al(OH)\,F_2} + 4\mathrm{OH^-} + 16\mathrm{SO_4^=}$
2) $Y\ \mathrm{MoF_3} + 2y\mathrm{OH_-} \rightarrow y\ \mathrm{Mo\,(OH)_3} + 2y\mathrm{F^-}$
3) $z\ \mathrm{Al_2O_3\ H_2O} + 2z\mathrm{H_2O} \rightarrow 2z\ \mathrm{Al(OH)_3} + 0.16z\ \mathrm{SO_4^=}$

By adding equations 1), 2) y 3) it is obtained:

4) $(x+z)\ \mathrm{Al_2O_3H_2O} + y\ \mathrm{MoF_3}+(4x-2y)\mathrm{F^-} - 0.16\ (x+z)\ \mathrm{SO_4^-} +$
$+\ 2(x+z)\ \mathrm{H_2O} \rightarrow 2\mathrm{Al(OH)F_2} + y\mathrm{Mo(OH)_2} + 2\ z\ \mathrm{Al\ (OH)\ 3} + (4x-2y)\mathrm{OH^-}$

After treating with H^+ the pH increases, the reaction being:

$$10^{-7} - 10^{-10}) \quad \text{x} \quad \frac{\text{Mo60}}{150\text{dm}^2} = 4{,}7 \text{ x } 10^{-4} \text{ gr equivalent/dm}^2$$

The analysis of the solution indicates consume values of Mo^{++} and F^- in 10mg/dm^2. Mo and F^- atomic weight is 95.94 approx. 96 and F^- 18,99 approx. 19 and it is obtained by

$$\frac{5{,}5 \text{ x } 10^{-5} \text{ x } 10^3}{4x-2y-0{,}32(x+z)} = \frac{10/96}{y}$$

$$\frac{10/19}{4x} = \frac{10/96}{y} \qquad y = 1.30$$

as $4x-2y -0.32 (x+z) = 0$ then $z = y$ assuming $x = 1$ and replacing equation 4)

$$5{,}20\ Al_2O_3{\cdot}H_2O + 1{,}3\ MoF_2 + 1{,}4\ F^- + 10.4\ H_2O\ \_\ {}_2Al(OH)F_2 +$$
$$+\ 1.3\ Mo(OH)_2 + 8.4\ Al(OH)_3\ 1.4\ OH^-$$

Cobalt New Sealing Solution	
Cobalt $Co^{++/+++}$	2.2 gr./l
Fluor F-	1.05 gr./l
pH	5.5
Temperature	27 ± 3°C
Time	8'-12'

Molybdenum New Sealing Solution	
Molybdenum $Mo^{6,5,4,3,2}$	2.5 gr./l
Flour F^-	1.15 gr./l
pH	5.4
Temperature	27 ± 3°C
Time:	8'-12'

COMPARED SEALING SOLUTIONS

Nickel Ni^{++}	2gr/l	Cobalt $Co^{++/+++}$	2.2 gr./l	Molybdenum $Mo^{6,5,4,3,2}$	2.5 gr./l
Fluor F^-	1 gr./l	Fluor	F^-1.05 gr/l	Flour F^-	1.15 gr./l
pH	5.6	pH	5.5	pH	5.4
Temperature	27 ± 3°C	Temperature	27 ± 3°C	Temperature	27 ± 3°C
Time	8'-12'	Time	8'-12'	Time	8'-12'

For the comparative sealing solution test temperature and time were maintained constant. Test ISO-3210 was applied for weight loss in mg/dm2 after anodizing. For cold sealing parameters and values had to be increased by 0.2 g/litre for cobalt and by 0.5 g/litre for molybdenum.

The cold sealing activator -free fluor-was increased from an initial concentration of 1.0 g/litre for fluor/nickel to values of 1.05 g/litre for fluor/cobalt and of 1.15 g/litre for fluor/molybdenum, that is, 0.05 g/litre more for fluor/cobalt and 0.15 g litre more for fluor molybdenum.

SEALING EXPERIMENTATION

ISO 3210 Test

Ni 11,5 mg/dm^2 F^- 13,6, 15± 1 mg/dm^2
Co 13,8 mg/dm^2 F^- 14,2, 15± 2 mg/dm^2
Mo 14,2 mg/dm^2 F^- 14,8, 15± 2 mg/dm^2

ISO 3210 Test

$NiF_2 + NiSO_y$
$CoF_2 + CoSO_y$
$MoF_2 + MoSO_y$

	Co *mg/dm^2*	*F^-* *mg/dm^2*	*ISO 3210* *mg/dm^2*
CoF_2+CoSO_4	10,2	14,1	14,0
$CoSO_4+HF$	11,0	13,0	17,0
$CoSO_4+N_aF$	20,3	28,5	30,0
CoAc+HF	10,2	7,5	27,0
CoAc+N_aF	13,0	13,0	35,0
CoAc+HF exc	11,0	38,0	59,0

	Mo *mg/dm_2*	*F^-* *mg/dm_2*	*ISO 3210* *mg/dm_2*
Mo F_2+$MoSO_y$	9,8	13,1	16,0
$MoSO_4$+HF	10,0	12,0	16,0
$MoSO_4$+NaF	18,0	29,0	25,0
MoAc+HF	10,0	9,5	24,0
MoAc+NaF	12,0	11,0	33,0
MoCab+HF exc	10,5	39,0	50,0

	NI *mg/dm_2*	*F^-* *mg/dm_2*	*ISO 3210* *mg/dm_2*
Ni F_2+$MoSO_y$	11.7	15.0	15.0
$NiSO_4$+HF	13.0	15.3	17.0
$NiSO_4$+NaF	18.2	23.5	26.0
NiAc+HF	11.5	8.7	25.0
NiAc+NaF	15.0	12.5	33.0
NiCab+HF exc	10.0	30.0	55.0

Aluminium panels of alloy 6065 were used and sulphuric acid concentrations were varied from 180 g/litre to 200 g/litre, the temperature changed and increased to 18°C ± 2°C to 25°C ± 2°C, without attacking the barrier layer. Current intensity was increased from 2.5 to 3 A/dm2 and the temperature maintained constant for 20 to 50 minutes.

Double Weight Loss Test ISO 3210.

Co acetate *5gr/l*	*Co camp* *13gr/dm^2*	*ISO Aw* *16mg/dm^2*
12.5	15.1	15.0
12.2	14.5	18.0
21.4	23.2	30.0
11.22	8.9	28.0
14.2	14.5	37.0
12.5	39.0	61.0

Mo sulphate *5gr/l*	*Mo camp* *12gr/dm^2*	*ISO Aw* *14mg/dm^2*
11.2	15.1	18.0
12.0	13.2	18.0
19.8	31.0	27.0
11.7	11.0	25.0
13.5	13.0	33.8
12.5	39.0	51.2

Ni acetate *5gr/l*	*Ni camp* *15gr/dm^2*	*ISO Aw* *18mg/dm^2*
11.6	15.0	16.3
13.2	15.5	17.0
18.0	23.9	26.1
11.3	9.2	25.1
15.1	12.5	33.1
10.1	31.0	55.0

For cobalt there are maximum differences of 2.3 mg/dm2 to 1 mg/dm2 in weight loss tests, while for molybdenum it was of 2 mg/dm2 to 1,5 mg/dm2 and for nickel of 0.1 mg/dm2 to 0.3 mg/dm2.

Anodizing

Sulphuric acid	180gr/l-200gr/lH_2SO_y
Temperature	18°C ± 2°C
Amperes	1.2 a 2.0 Am/dm2
Time	20' a 50'
Sulphuric Acid	150gr/l-250gr./l H_2SO_y (without stain)
Temperature	13°C ± 27°C
Amperes	0.8 a 3.5 Am/dm2 (w/out barrier layer's burn)
Time	20' a 50'

All parameters were at 20°C to 50°C. temperature and alloy 6065 used for aluminium sample panels.

TABLE 1

Structural Parameters From X-Ray Diffraction Patterns Of Anodized And Sealed Al Samples

SEALING PROCESS	***Normal rotation*** $\ddot{A}I_r^m$ ***at*** $d^=.36nm$			***Tangential scattering curve*** $\ddot{A}I_r^m$ ***at*** $S^=.28nm^{-1}$			$\ddot{A}_r/dS$ ***at*** $S^=.45nm^{-1}$		
	Ni	***Co***	***Mo***	***Ni***	***Co***	***Mo***	***Ni***	***C0***	***Mo***
Cold Sealing	0.35	0.36	0.37	1.55	1.54	1.56	5.40	5.50	5.50
Bath A	0.34	0.35	0.35	1.56	1.57	1.57	5.40	5.50	5.50
Bath B	0.29	0.30	0.30	1.56	1.57	1.57	5.60	5.65	5.65
Medium Sealing	0.39	0.39	0.39	1.92	1.92	1.92	7.00	7.00	7.00
Hot Sealing	0.40	0.40	0.40	1.32	1.32	1.32	4.90	4.90	4.90

Samples after Weathering: 9 month - Industrial Atmosphere

	Ni	Co	Mo	Ni		Co	Mo	Ni	Co	Mo	
Cold Sealing	0.50	0.50	0.50		0.75	0.75	0.75		3.00	3.00	3.00

$I_r = I/I_{book\,ground}$ $\quad I_r^m = I^{maximum}/I_{b2}^{-1}$

TABLE 2

Knoop Hardness Values, 26 g Load With Indentation At 5μm from the Al/Al_2o_3 Interface

Sealing Process	Knoop Hardness Number **Ni**	**Co**	**Mo**	
Cold Sealing	390±25	388±25	387±25	Variance in Co/Mo
Medium Sealing	350±25	345±25	340±25	Variance in Co/Mo
Hot Sealing	--	--	--	
Weathered Cold Sealing	220±40	215±40	215±40	Without variance
Weathered Hot Sealing	--	--	--	

MILITARY SPECIFICATIONS

Seal Process Specifications:

1. Class I (all types). Undyed coatings-hot potassium or sodium dichromate, nickel or cobalt acetates, D.I. water or other suitable solutions. Specific conditions apply.
2. Class II (all types) Dyed coatings -Hot nickel or cobalt acetate, D.I. water, duplex (nickel acetated, then sodium dichromate) other suitable solutions, specific conditions apply.
3. Type III –Only when required by contract; use Calls I.
4. ***Standard Test** Procedures*: Salt Spray ASTM B-117; 336 hours required.

SAE, AEROSPACE MATERIAL SPECIFICATIONS

Scope:

1. Seal requirements for the following:
    - AMS 2471E, Undyed Sulphuric Coatings.
    - AMS 2472D, Dyed Sulphuric Coatings.
    - MS 2470J, Chromic acid Coatings.
2. Corrosion resistance and paint topcoat are primary goals of process specifications.
3. Conformance is voluntary.
4. Aerospace companies require specific in-house standards.

MILITARY SPECIFICACIONES

Scope

1. Seals for MIL-1-8625F anodised finishers; Type 1, 1B, 1C, 11, 11B. No seals for Type III (HC unless specified.
2. Seals for MIL-A-63576A (AR) lubricative anodic coatings. Types I, II, III. These are PTFE processes.
3. Conformance to specifications is mandatory.

AUTOMOTIVE STANDARDS

Scope

1. All automotive manufactures prove specific in-house specifications and standards.
2. Nickel acetate pre-seal followed by hot D.I water is the predominant process specification

SAE, AEROSPACE MATERIAL SPECIFICATIONS

Seal Process Specifications

- ➢ Water Operating conditions specified.
- ➢ Sodium or potassium dichromate solutions; conditions specified.

- Nickel or cobalt acetates solutions specified.

Test Procedures and Requirements

- Salt spray, ASTM B-117 336 hours for all specifications

Silicate Sealing

One of the possible mechanisms for silicate sealing is based on the formation of silica. The optimum ratio for soda-silicate should be around 1:3.3 (Na_2O: SiO_2). A typical procedure for silicate sealing is boiling the parts for approximately 30 minutes in a solution containing 5% sodium silicate. The effect of harness and wear resistance is reported to be minimal, mainly because no boehmite is formed.

Miscellaneous Sealing Methods

- Sealing by electrode position
- Fluorpolymers impregnation

The impregnation fluorpolymers, are called organic or impregnation sealing and leave non-wetting surface. The working temperature is about 30-40°C, the concentration is 15-30%, the pH is alkaline 8.5-9.00, the contact time is 30-40 sec, and dries at 75°C.

TABLE 3.
COMPARATIVE RESULTS FOR WEIGHT LOSS ASTM B-680

Post Treatment	Sealing Time	Weight loss (mg/dm^2)
Hot water seal	15 min	78
	30 min	55
	45 min	45
	60 min	45
	120 min	30
Medium temperature seal	10 min	420
	30 min	120
	60 min	15
Dichromate seal	5 min	800
	10 min	700
	15 min	400
	20 mn	200
Medium temperature silicate seal	90 sec	800
Organic sealer	contact	0
Medium temperature seal	60 min	100
High speed nickel acetate	30 min	120

TABLE 4
HARDNESS TESTING - ISO 4516

Post Treatment	Sealing Time	Micro Substrate	hardness (25 p) side<------------>	HV (n=8) surface oxide
Hot water seal	15 min	430	430	330
	60 min	500	430	425
Medium temperature seal A	60 min	500	500	400
Medium temperature silicate seal	90 sec	450	440	440
Organic sealer	contact	460	460	410
Medium temperature seal B	60 min	460	480	450
High speed nickel acetate	30 min.	425	410	450
Cold sealing Ni	10 min	450	400	300
Cold seal CO	10 min	400	395	275
Cold seal Mo	10 min	390	375	250

It can be see there is little difference on micro-hardness, which is not determinant.

For each parameter three tests were made in the laboratory and observed and compared sealing time and the respective micro-hardness as shown in Table 4. All work with fluorpolymers took place at a company near Barcelona, Spain. The company is specialized in hard anodizing.

The organic coating being used as mechanism, such a as silicate sealing, are very important for new physical coatings and finishings on aluminium anodising and particularly for hard anodising.

Organic coated aluminium is used for home electric appliances, precision mechanics, textile industry, and automation, etc. since it has good corrosion resistance, and easy to produce economically.

Required properties of organic coated aluminium for home electric appliances are anti-fingerprinting and self-lubricant sheet.

Anti-fingerprinting sheet has good performance against anti-finger printability and self-lubricant sheet had excellent formability without press oil. But thin organic coated aluminium does not have enough resistance and conductibility characteristics.

Composition of resin solution will affect the quality of the above characteristics.

In this research, the effect of resin and additives for thin organic coated aluminium was investigated.

Experimental Method

Resin Coating

Chromate electro galvanizing aluminium was used as a substrate for resin coating.

Chromate coating weight was 15-20 mg/m^2 and that of aluminium 10-15 m^2. Resin was coated using a bar coater and then specimen was baked at moderate temperature using the cabinet type drying oven. The dry film thickness was adjusted by various size of bar coater.

Measurement of Physical Properties

Anti-finger printability was estimated by colour difference value (ΔE) between before and after vaseline coating. Corrosion resistance was measured with plate specimens. Then corrosion test was performed with salt spray tester.

The dynamic friction coefficient was measured using a draw bead tester and calculated using the following equation.

$$\mu = \frac{Fd - Rd}{Fc \times \pi}$$

Fc = fixed bead clamping force
Fd = Fixed bead drawing force
Rd = roller bead drawing force

The test conditions was as follows:
- drawing speed: 500 mm/min
- bead radius: R5,25

Interlaminar Resistance

To compare conductibility of organic coated aluminium, interlaminar resistance was measured. We measured current value after making a firm contact of an electrode with specimen at a constant pressure.

Standard pressure: $2N/mm^2 \pm 5\%$
Experimental voltage: 1 V
Electric power: 0.5-15A
R = A (1/I-1)

Heat Resistance

Although silica improves corrosion resistance, it hampers formability of organic coated aluminium. To evaluate the effect of silica, the amount of additive silica in resin was varies without adding any other additive. The thickness of coating film was chosen as 3µm in order to minimize the effect of aluminium substrate.

Fig. 1 shows the effect of silica on friction coefficient. As the amount of silica increases, friction coefficient increases too. The result suggests that addition of silica would degrade formability. SO – S4 means the ratio of solid content of resin to silica and it varies form 0-20 phr.

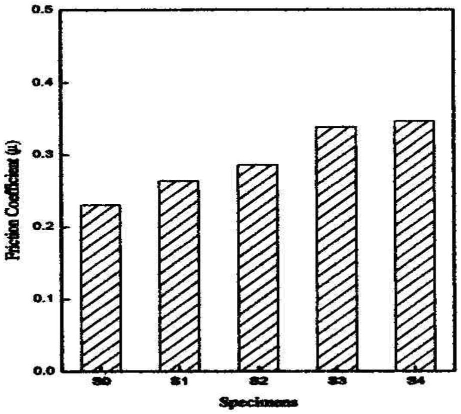

Figure 1. Friction coefficient with silica content.

Without silica, red rust was observed all over the surface. However, in case of S4, white rust was observed only at limited area. This result means that silica in resin improves corrosion resistance of organic coated aluminium.

Fig. 2 shows ante-fingerprinting property of organic coated aluminium as a function of silica content.

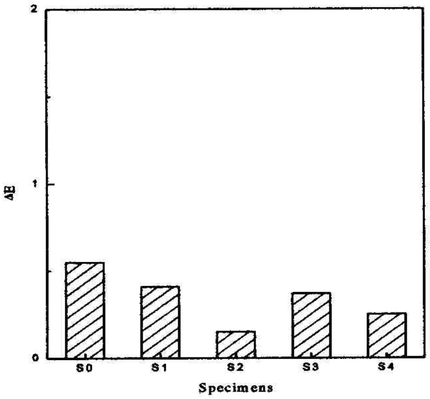

Figure 2. Anti-finger property with silica content.

Since hydrophobic silica particles are evenly dispersed in organic coated aluminium regardless of silica content anti-fingerprinting property does not seem to be significantly affected by the amount of silica in resin. The even distribution of silica was confirmed by surface morphology and Si mapping of samples.

Heat resistance Improvement

To improve heat resistance of organic coated aluminium sheet, the effect of heat resistant additives on heat resistance was scrutinized. Table 5 shows the variation of chemicals composition of solution by changing ratio of the base resin (A) to the heat resistant resin (B).

Table 5. Composition Of Resin Solution

Item	Base Resin (A)	H.R. Resin B)	Hardener	Water
M1	15	141	2.52	18.21
M2	12	21	1.95	15.06
M3	9	37	1.25	9.07

Fig. 3 shows the weight loss data of each resin with different chemical composition during TGA tests.

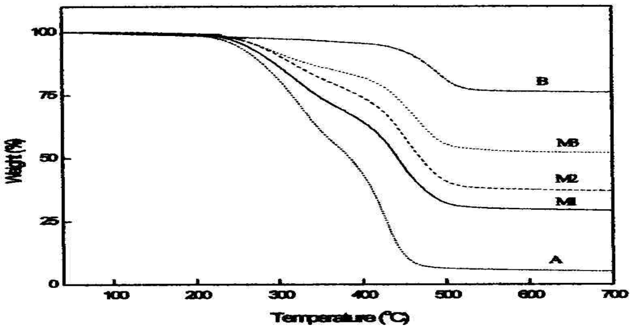

Figure 3.TGA results for various resins

The results show that heat stability is superior at following order: B, M1, M2, M3, A. Heat stability of the heat resistant resin B is so superior to that of resin A that weight loss is only 16.5% up to $700^{0.}$

Fig. 4 shows variation of whiteness after exposing each sample with different solution composition at $185^{0.}$

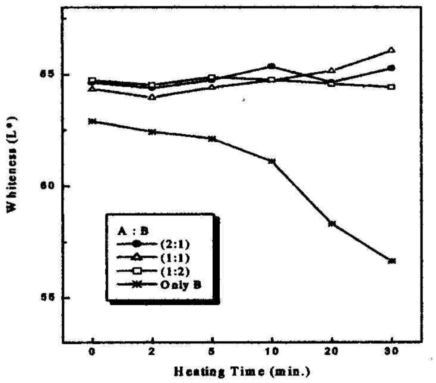

Fig. 4. Changes of whiteness for various resins

When adding heat resistant resin, no colour change was observed. However, base resin shows a sudden decrease in whiteness. Although the heat resistant resin improves heat resistance of organic coated aluminium sheet, with the heart resistant resin, white spots were observed on the sample surface. Since white spot degrade the surface quality of organic coated aluminium, there should be a further study to develop a way to remove the white spots.

Variation of friction coefficient, as a function of the heat resistant resin is in Fig. 5.

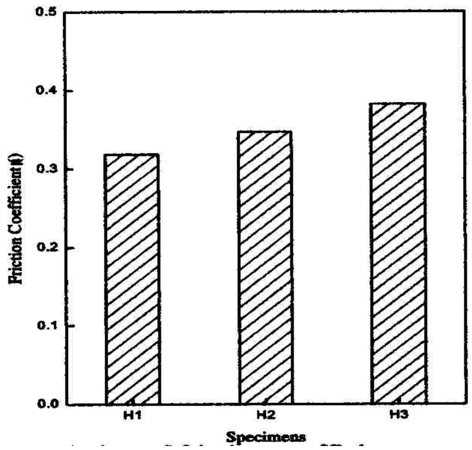

Figure 5.Variation of friction coefficient.

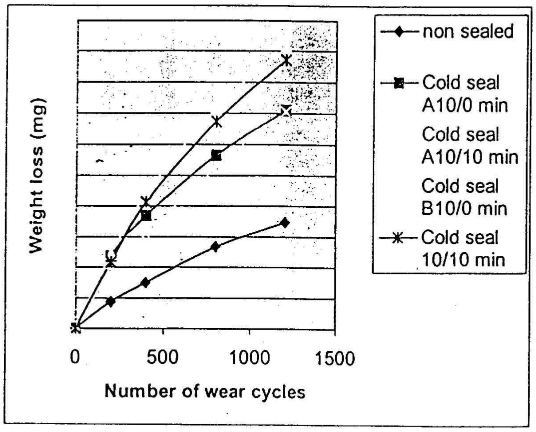

Figure 6. Abrasive wear resistance cold sealed coatings, artificially aged

Although friction coefficient of the sample with heat resistant resin is bigger than that of base resin, ratio of heat resin to the base resin does not affect significantly. The increase of friction coefficient seems to be related to the existence of silica, which is in the heat resistant resin.

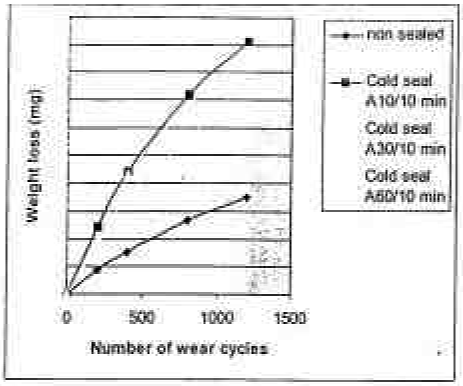

Fig. 7. Abrasive wear resistance cold sealed coatings, with and without artificial ageing

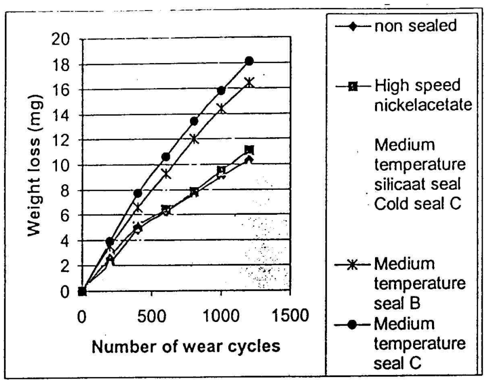

Fig. 8. Abrasive wear resistance hard anodic coatings with different sealing methods.

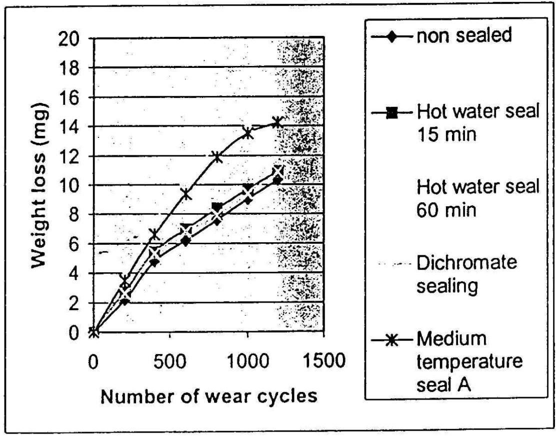

Fig. 9. Abrasive wear resistance hard anodic coatings with conventional sealing methods.

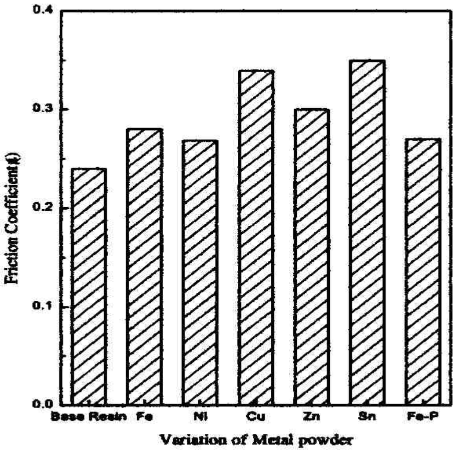

Figure 10. Comparison of friction coefficient

Metal Powder

During welding organic film layer is carbonised and as a result, there are problems of low longevity of welding electrode and poor surface appearance. To solve the above welding problems, we added metal powder to organic coating layer to lower electric resistance of organic film layer. The metal powders used were Fe, Ni, Cu, Zn, Sn, Fe-P and their size was about 1-8 μm. When adding metal powders, friction coefficient increased slightly. (Fig. 10)

This results suggest that metal powders seem to degrade formability of organic coated aluminium. Table 2 shows interlaminar resistance of coating layer with 10-25wt% metal powders. As metal powders were added, its interlaminar resistance decreased significantly. Therefore, addition of metal powder would improve weldability of organic coated aluminium.

Table 6.
Electric resistance of metal powder

Specimens	Current (A)	Resistance
Fe (10%)	0.99	0.50
Ni (10%)	0.98	0.04
Cu (10%)	0.89	0.50
Zn (10%)	0.72	4.20
Sn (10%)	0.80	2.50
Fe-P (10%)	0.96	0.03
Base resin	0.085	75.25

However, addition of metal powder makes solution unstable. That metal powder is settled at the bottom and solution becomes gel. Therefore, there should be found an appropriate solution to resolve the above problem if we want to use metal powder added to organic coated aluminium.

CONCLUSIONS

1. The corrosion resistance was improved by the addition of silica, but silica does not help the lubricating action.
2. Addition of metal powder in resin gives a positive effect on the conductibility whereas it may degrade de resin solution stability.
3. Although addition of heat resistant resin has improved heat resistance or organic coated aluminium, it deteriorates surface appearance
4. Cobalt and molybdenum sealing are perfectly valid and have less environmental impact than nickel, but they need further laboratory testing.
5. ISO 3210 tests are perfectly valid for cobalt/molybdenum
6. Knoop's hardness values are perfectly valid as shown in the comparative studies on cold and medium sealing.
7. Water sealing assures a similar Rcorr to dichromate sealing but better Rwaste and allow surface coloration.
8. The fluorpolymers sealing assures a correct Rcorr and good Rwaste.
9. Depending on the fluorpolymers some can only coat the surface and some coat and penetrate into the surface.

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