BONDERITE®

LOW FLUORIDE CONVERSION COATING PROCESS FOR ALUMINUM PROFILE WITH WEATHER RESISTANT POWDER

TAO YANG

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Henkel

Henkel Adhesive Technologies

Abstract

The affection of aluminum alloy treated with BONDERITE M-NT 4830N7 process and then painted with AKZO powder(C56209S2VP(D2015FC)) on High-pressure boiling test and Acetic Acid Salt Spray Test (AASS) performance with different degreasing, they had been investigated by DOE experiments. They are BONDERITE C-IC 60003 which contains high fluorine ion and BONDERITE C-AK 34 with Deoxidizer which has low fluorine ion. The test results showed that the low fluorine process could achieve the same level as high fluorine process. The whole studies offer clear pictures of Boiling test and AASS test performance with different pretreatment conditions.

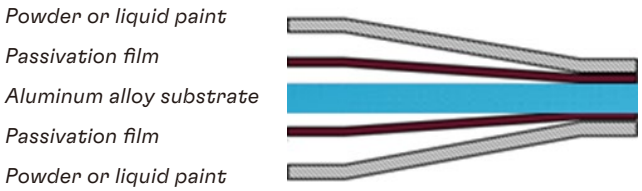
Introduction

Henkel's Cr-free passivation pretreatment process for aluminum is commercialized about thirty years, it's an environment friendly technique dues to Cr-free. Most applications use acid degreasing like BONDERITE C-IC 60003, the wastewater contains about 50 ppm fluorine ions, most local governments require wastewater that can be discharged to contain less than 10 ppm of fluorine ions, so the customers need to treat fluorine ions, they usually use $CaCl_2$ to treat fluorine ions, approximately equivalent to 5% of the passivation pretreatment fee. but some local governments require wastewater that can be discharged to contain less than 2 ppm of fluorine ions, it very difficult to meet the requirements. BONDERITE C-AK 34 is Alkali degreasing, the process with Deoxidizer has fluorine ion below 10 ppm, so if Alkali degreasing process could achieve the same level as high fluorine process, it will save on wastewater treatment costs and easy to meet the government requirement that fluorine ions are less than 2 ppm during wastewater discharge, this process will be more acceptable to customers.

Definitions

a) Aluminum alloy Painting system

For improving the anti-corrosion performance to prolonging the usage life of working parts, aluminum alloys parts are usually treated with pretreatment process and then painted with powder coating or liquid paint. And the anti-corrosion performance is determined by the pretreatment process and powder or liquid paint painting process.



Picture: Aluminum alloy painting system

b) Aluminum alloy passivation film

Hexavalent chromium oxide film layer mainly consist of Oxide hydrates of Cr (VI), Cr (III) und Al (III), trivalent chromium oxide film layer mainly consist of Oxide hydrates of Cr (III) und Al (III) and Phosphates of Cr (III). BONDERITE M-NT 4830N7 is a Cr-free passivation product, it mainly consists of Zr oxyfluoride, hydroxides and fluorides of aluminum, organic. Parts of the reaction are as below:

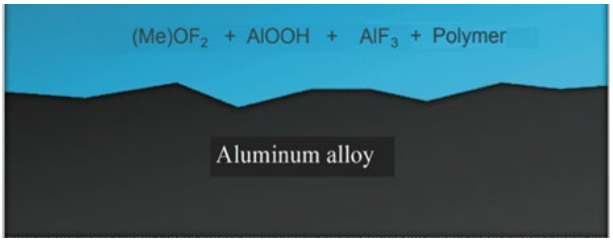
(1) indicates the corrosion reaction, HF is consumed which makes chemical equilibrium of (2) moving to right, and the coating layer is gradually formulated on the surface with the form of $ZrOF_2$, AlF_3, $Al(OH)_3$ and Polymer .

Etching reaction:

$Al + 3H^+ \rightarrow Al^{3+} + H^2$

Formation of membrane layer:

$H_2ZrF_6 + Al^{3+} + H_2O$ + Polymer $\rightarrow ZrOF_2 + AlF_3 + Al(OH)_3$+ Polymer

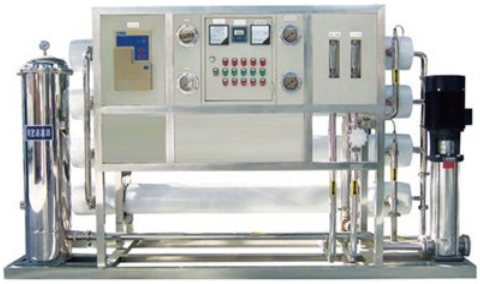


Picture: 4830N7 reaction (Me is Zr here)

c) Tap water/DI water (RO water)

Tap water usually contains various ions like Ca^{2+} and Mg^{2+} and so on, the concentration of these ions is different in different regions, and conductivity could be used to indicate the level of concentration of ions. The higher the conductivity, the higher concentration of ions in water, and these ions bring negative impact on the formation of passivation.

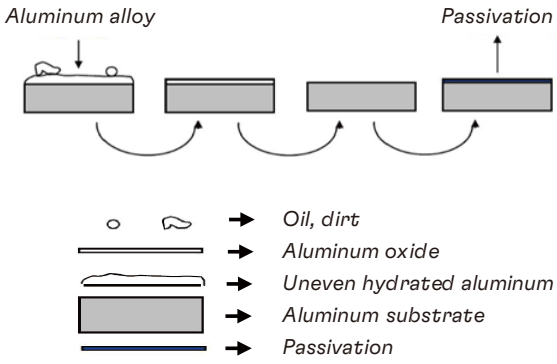
DI water or RO water is produced by tap water through ion-exchange equipment or reverse osmosis equipment. The conductivity of DI water or RO water is very low (10μs/cm approximately), and it can provide positive affect on the formation of passivation.



Picture: RO water equipment

d) Cr-free Pretreatment process of aluminum alloy

The pretreatment process has two kinds of degreasing: Acid degreasing and Alkali degreasing, the purpose is to remove oil、dirt、aluminum oxide and uneven hydrated aluminum. Under the same amout of corrosion, the grains of aluminum alloys are finer and the appearance is more uniform than those treated with alkali degreasing.



Picture: Cr-free passivation of aluminum alloy

e) Etching amount test

Aluminum alloys need to remove oil stains and oxide scales before applying passivation films, Qualicoat certificate authority require etching amount above 1 g/m^2, it's used to ensure that conventional oil stains and oxide scales can be completely removed.

Etching amount test method is as below:

(1) Wipe the surface of the test panel with cotton dipped in alcohol.

(2) Dry the panel at around 40°C and weight it, record as M1.

(3) Treat the test panel with pretreatment process.

(4) Dry the panel at around 40°C and weight it, record as M2.

(5) Count: (M2-M1)/m^2

f) High-pressure boiling test

High-pressure boiling test is used to test the adhesion performance of aluminum panels after pretreatment and spraying, the test method is as below:

Inject DI water into a pressure cooker to a depth of about 80 mm, place a sample of about 50 mm in length perpendicular to the water. The sample should be 10 mm below the water surface, but not in contact with the bottom of the container. Heat it to a pressure of 0.1 MPa ± 0.01 MPa and maintain a constant pressure for 1 hour. Remove and dry the sample, visually inspect the changes in the film surface after the test and conduct an adhesion test within 5 minutes of removing the sample. The requirement is that there should be no bubbles in the film layer after the test, and crosscut the film layer, the powder should not peel off after sticking the tape.

Determination of adhesion test level:

Level 0, no detachment.

Level 1, with only small pieces of paint peeling off at the intersection of the cut, and the actual damaged area in the marked area is less than or equal to 5% of the coating.

Level 2 indicates partial peeling along the edge of the incision, with a peeling area between 5% and 15%.

Picture: Pressure steam sterilizer and Crosscut Tester

g) AASS (Acetic Acid Salt Spray Test)

AASS is a lab method to evaluate the anti-corrosion performance of painting system. Normally, the sprayed solution is 5% ± 0.5% (w/w) NaCl, use acetic to adjust PH to 3.1-3.3, painted samples are placed in salt spray chamber sprayed under a certain pressure at 35°C ± 2°C, The salt fog will fall and deposit on the samples which grooved ✖, then observe and evaluate the corrosion degree after a period.

Qualicoat certificate authority require length of the biggest infiltration: L<3mm, surface infiltration: <16mm^2/10cm.

Picture: AASS TEST Salt fog box

Pretreatment Process Design

Design 5 kinds of Pretreatment process, six aluminum alloy panels are selected for each pretreatment process, painted with AKZO powder (C56209S2VP(D2015FC)), then take 3 panels to do the High-pressure boiling test and 3 panels to do AASS test.

a) Pretreatment process with acid degreasing

Currently, most of aluminum alloy line using the following process:

Acid degreasing – Tap water – Tap water – DI water – Passivation

No	Stage	Chemical	Parameters
1	Degreasing	BONDERITE C-IC 60003 BONDERITE C-AD 1025S	Free Acid (FAL)=8 Time=6min 21.2°C Etch amount 1.85g/m^2 (made up by tap water)
2	Rinse	Tap water	Cond=186μs/cm Time=1min
3	Rinse	Tap water	Cond=186μs/cm Time=1min
4	Rinse	DI water	Cond=6μs/cm Time=1min
5	Passivation	BONDERITE M-NT 4830N7	Conc=0.1% PH=3.52 Time=1min (made up by DI water)

Use this process, the wastewater contains about 50 ppm fluorine ions, most local governments require wastewater that can be discharged to contain less than 10 ppm of fluorine ions, approximately equivalent to 5% of the passivation pretreatment fee.

b) To reduce fluorine ion, Alkali degreasing to instead Acid degreasing, the process as below:

Alkali degreasing – Tap water –Tap water – Deoxidizer – Tap water – DI water – Passivation

No	Stage	Chemical	Parameters
1	Degreasing	BONDERITE C-AK 34 BONDERITE C-AD 1025S	Free Alkali (FAL)=10 Time=6min 21.2°C Etch amount 2.12g/m2 (made up by tap water)
2	Rinse	Tap water	Cond=186μs/cm Time=1min
3	Rinse	Tap water	Cond=186μs/cm Time=1min
4	Deoxidizer	BONDERITE C-IC 395H	Free Acid (FAL)=10.2 Time=1min
5	Rinse	Tap water	Cond=186μs/cm Time=1min
6	Rinse	DI water	Cond=6μs/cm Time=1min
7	Passivation	BONDERITE M-NT 4830N7	Conc=0.1% PH=3.52 Time=1min (made up by DI water)

In this process, BONDERITE C-IC 395H is used to neutralizing alkali and aluminum hydroxide for its low cost, but for AASS test is not the best choice, and the surface of aluminum alloy exists uneven corrosion situation.

c) To improve the AASS test performance, replace BONDERITE C-IC 395H with BONDERITE M-NT 2040, the process as below:

Alkali degreasing – Tap water –Tap water – Deoxidizer – Tap water – DI water – Pasivation

No	Stage	Chemical	Parameters
1	Degreasing	BONDERITE C-AK 34 BONDERITE C-AD 1025S	Free Alkali (FAL)=10 Time=6min 21.2°C Etch amount 2.12g/m^2 (made up by tap water)
2	Rinse	Tap water	Cond=186μs/cm Time=1min
3	Rinse	Tap water	Cond=186μs/cm Time=1min
4	Deoxidizer	BONDERITE M-NT 2040	Free Acid (FAL)=12.1 Time=1min
5	Rinse	Tap water	Cond=186μs/cm Time=1min
6	Rinse	DI water	Cond=6μs/cm Time=1min
7	Passivation	BONDERITE M-NT 4830N7	Conc=0.1% PH=3.52 Time=1min (made up by DI water)

In this process the surface of aluminum alloy also exists uneven corrosion situation.

d) To improve the surface of aluminum alloy, add acid activation before the alkali degreasing, the process as below:

Acid activation– Alkali degreasing – Tap water –Tap water – Deoxidizer – Tap water – DI water – Passivation

No	Stage	Chemical	Parameters
1	Acid activation	BONDERITE C-IC 60003	Conc=0.05% Cond=1086μs/cm (made up by tap water)
2	Degreasing	BONDERITE C-AK 34 BONDERITE C-AD 1025S	Free Alkali (FAL)=10 Time=6min 21.2°C Etch amount 2.12g/m^2 (made up by tap water)
3	Rinse	Tap water	Cond=186μs/cm Time=1min
4	Rinse	Tap water	Cond=186μs/cm Time=1min
5	Deoxidizer	BONDERITE C-IC 395H	Free Acid (FAL)=10.2 Time=1min
6	Rinse	Tap water	Cond=186μs/cm Time=1min
7	Rinse	DI water	Cond=6μs/cm Time=1min
8	Passivation	BONDERITE M-NT 4830N7	Conc=0.1% PH=3.52 Time=1min (made up by DI water)

In this process, BONDERITE C-IC 395H is used to neutralizing alkali and aluminum hydroxide for its low cost, but for AASS test is not the best choice, the surface of aluminum alloy is normal.

e) To improve the AASS test performance, replace BONDERITE C-IC 395H with BONDERITE M-NT 2040, the process as below:

Acid activation– Alkali degreasing – Tap water –Tap water – Deoxidizer – Tap water – DI water – Passivation

No	Stage	Chemical	Parameters
1	Acid activation	BONDERITE C-IC 60003	Conc=0.05% Cond=1086μs/cm (made up by tap water)
2	Degreasing	BONDERITE C-AK 34 BONDERITE C-AD 1025S	Free Alkali (FAL)=10 Time=6min 21.2°C Etch amount 2.12g/m² (made up by tap water)
3	Rinse	Tap water	Cond=186μs/cm Time=1min
4	Rinse	Tap water	Cond=186μs/cm Time=1min
5	Deoxidizer	BONDERITE M-NT 2040	Free Acid (FAL)=12.1 Time=1min
6	Rinse	Tap water	Cond=186μs/cm Time=1min
7	Rinse	DI water	Cond=6μs/cm Time=1min
8	Passivation	BONDERITE M-NT 4830N7	Conc=0.1% PH=3.52 Time=1min (made up by DI water)

In this process, the surface of aluminum alloy is normal.

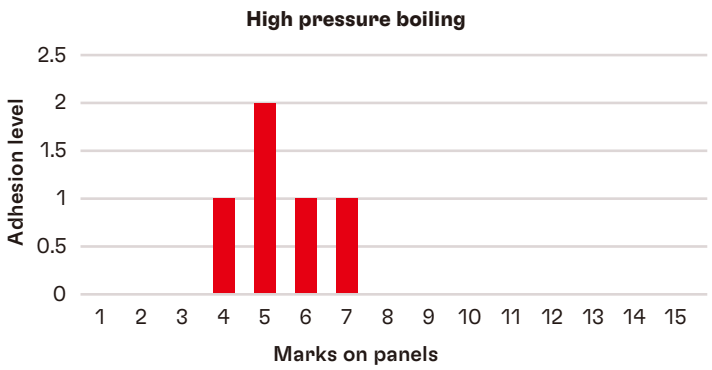
Results and Discussion

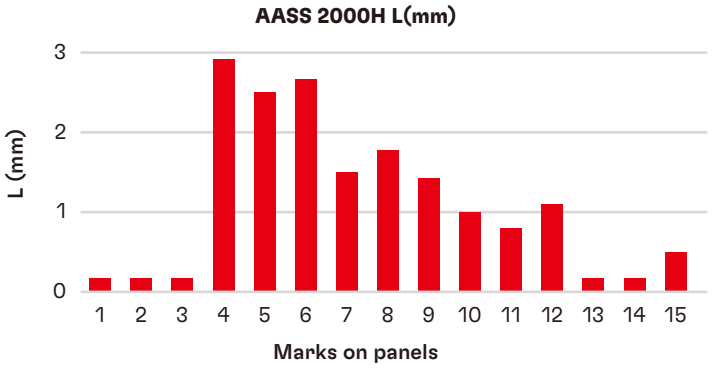
The detailed parameters for the test in this paper are shown as below:

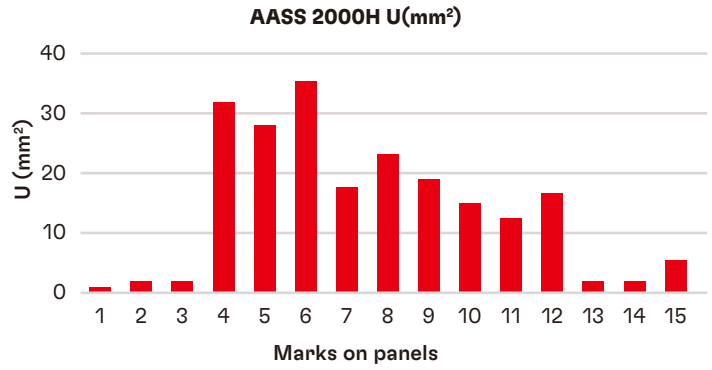
a) Test result record

Process	Marks on panels	Powder thickness(um)	High pressure boiling level 0 passed	AASS 2000H L(mm)<3	AASS 2000H U(mm²)<16
a	1	68-82	0	0.2	1
	2	60-77	0	0.2	2
	3	66-71	0	0.2	2
b	4	61-78	1	2.8	32
	5	72-87	2	2.4	28
	6	65-89	1	2.6	35
c	7	78-86	1	1.5	18
	8	63-81	0	1.7	23
	9	66-79	0	1.4	19
d	10	68-83	0	1.0	15
	11	71-89	0	0.8	12
	12	76-91	0	1.1	16
e	13	62-75	0	0.2	2
	14	61-82	0	0.2	2
	15	71-90	0	0.5	5

b) Test result

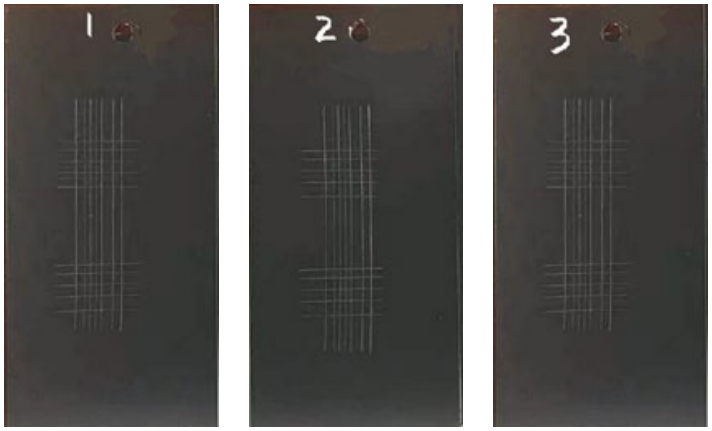


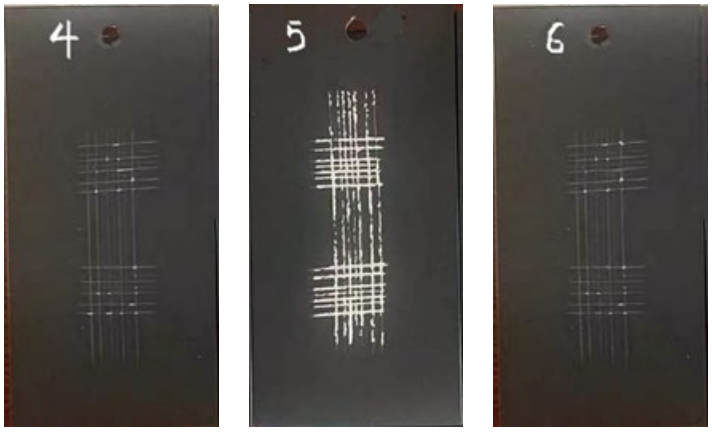


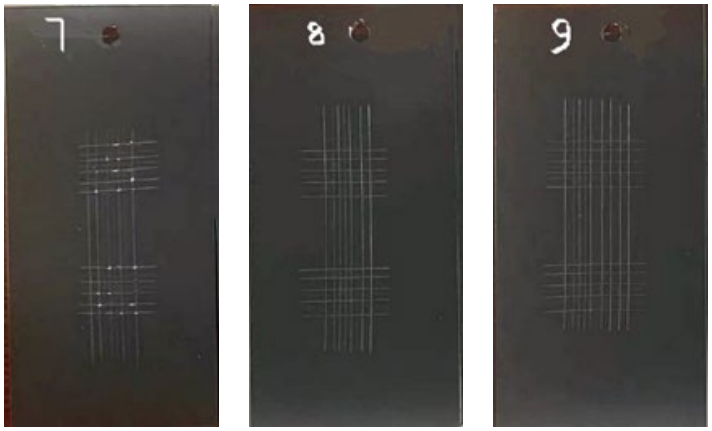


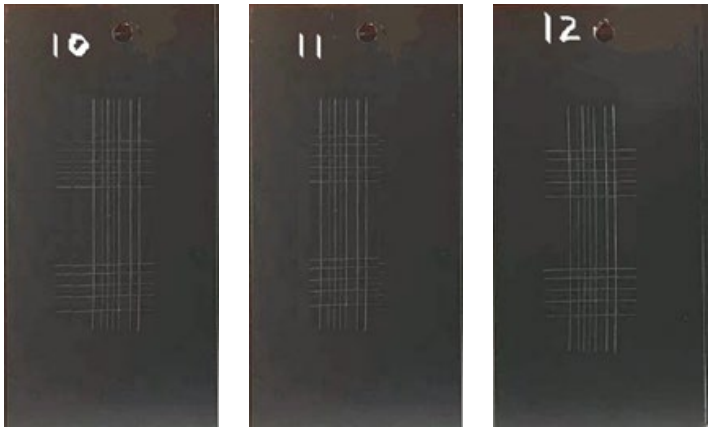
c) Test pictures

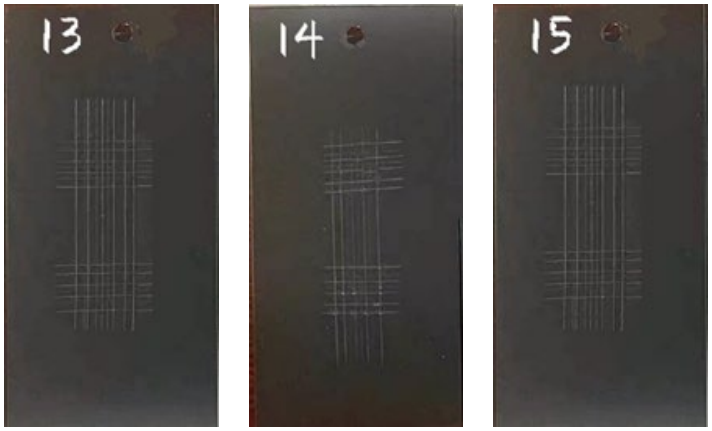
The picture of High pressure boiling test as below:



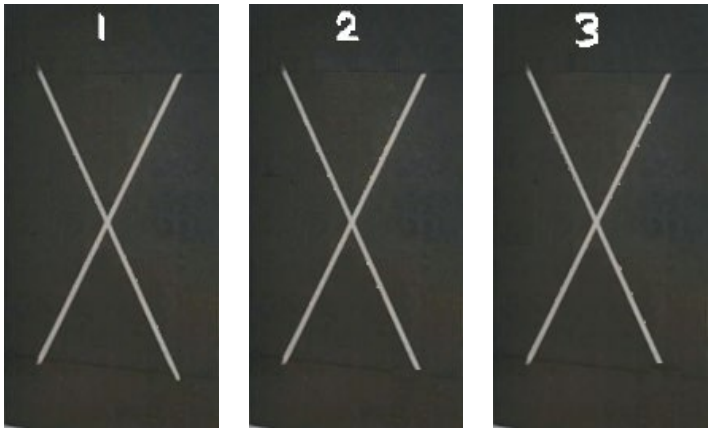


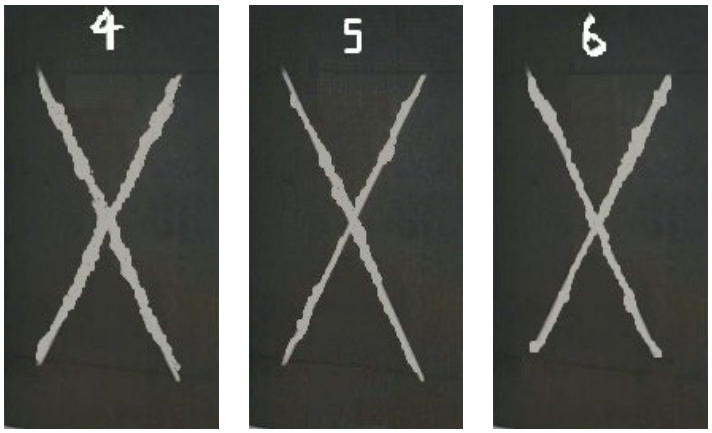


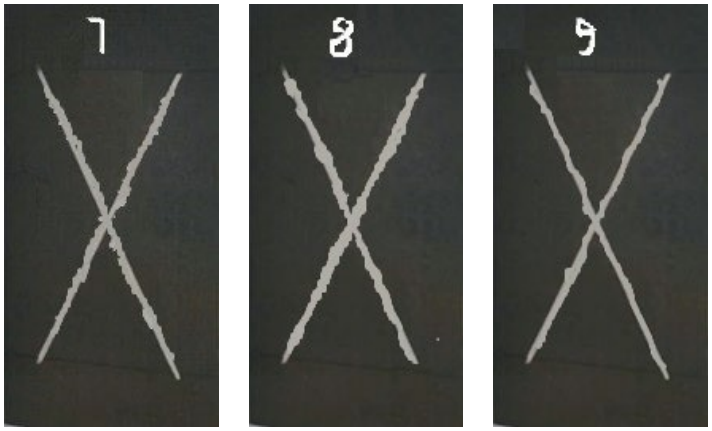


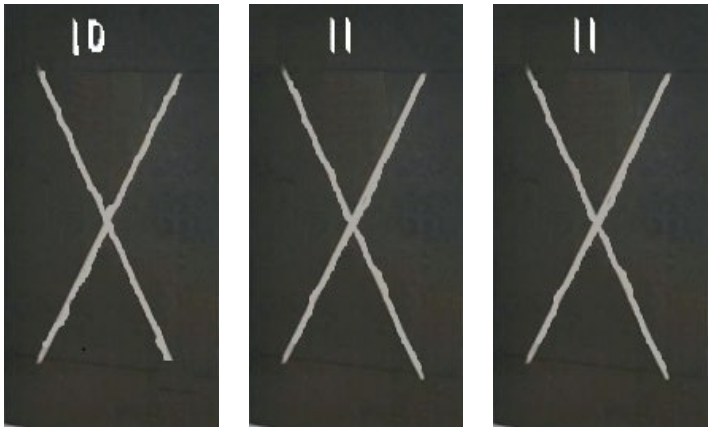


The picture of AASS test as below:









Conclusions

a) The performance of High-pressure boiling test:
1. The pretreatment process with acid degreasing all passed this test.
2. The performance of using alkali degreasing was worse than acid degreasing, 3 panels using BONDERITE C-IC 395H for deoxidization all failed the test, 2panels were level 1 and 1 panel was level 2.
Using BONDERITE M-NT 2040 for deoxidization better than BONDERITE C-IC 395H, 2 panels passed and 1 panel was failed with level 1.
3. The pretreatment process adding acid activation before alkali degreasing all passed this test, no only with BONDERITE M-NT 2040 for deoxidization passed the test, with BONDERITE C-IC 395H for deoxidization also passed the test.
b) AASS performance test:
1. The pretreatment process with acid degreasing all passed this test, it's the best process, the average of the biggest infiltration length was 0.2 mm, the average of the surface infiltration was 1.67 mm^2/10 cm.
2. the performance of using alkali degreasing was worse than acid degreasing, using BONDERITE M-NT 2040 for deoxidization did better than BONDERITE C-IC 395H.
BONDERITE C-IC 395H for deoxidization: the average of the biggest infiltration length was 2.6 mm; the average of the surface infiltration was 31.67 mm^2/10 cm.
BONDERITE M-NT 2040 for deoxidization: the average of the biggest infiltration length was 1.53 mm; the average of the surface infiltration was 20.00 mm^2/10 cm.
3. The process of adding acid activation before alkali degreasing all passed this test.
BONDERITE C-IC 395H for deoxidization: the average of the biggest infiltration length was 0.97 mm; the average of the surface infiltration was 14.33 mm^2/10 cm.
BONDERITE M-NT 2040 for deoxidization: the average of the biggest infiltration length was 0.3 mm; the average of the surface infiltration was 3.00 mm^2/10 cm.
c) The best pretreatment process with low fluorine as below: Acid activation(60003)– Alkali degreasing(34) – Tap water –Tap water – Deoxidizer(2040) – Tap water – DI water – Passivation(4830 N7).
In term of high-pressure boiling test and AASS test, after spraying AKZO weather resistant powder on aluminum alloy using this low fluorine process, it's performance can reach the level as high fluorine process.

References

a) All panels were painted with AKZO powder (C56209S2VP(D2015FC)) and tested in the lab of GuangYa aluminum Co.Ltd.