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Patrice Brun. Influence of impregnation parameters on structure and properties of plasma sprayed alumina coating impregnated by aluminium phosphate.. matériaux 2022, Oct 2022, Lille, France. 2022. cea-04742477

HAL Id: cea-04742477

<https://cea.hal.science/cea-04742477v1>

Submitted on 17 Oct 2024

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INFLUENCE OF IMPREGNATION PARAMETERS ON THE STRUCTURE AND PROPERTIES OF PLASMA SPRAYED ALUMINA COATING IMPREGNATED WITH ALUMINUM PHOSPHATE

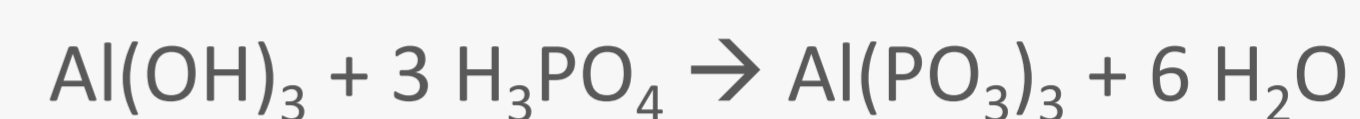
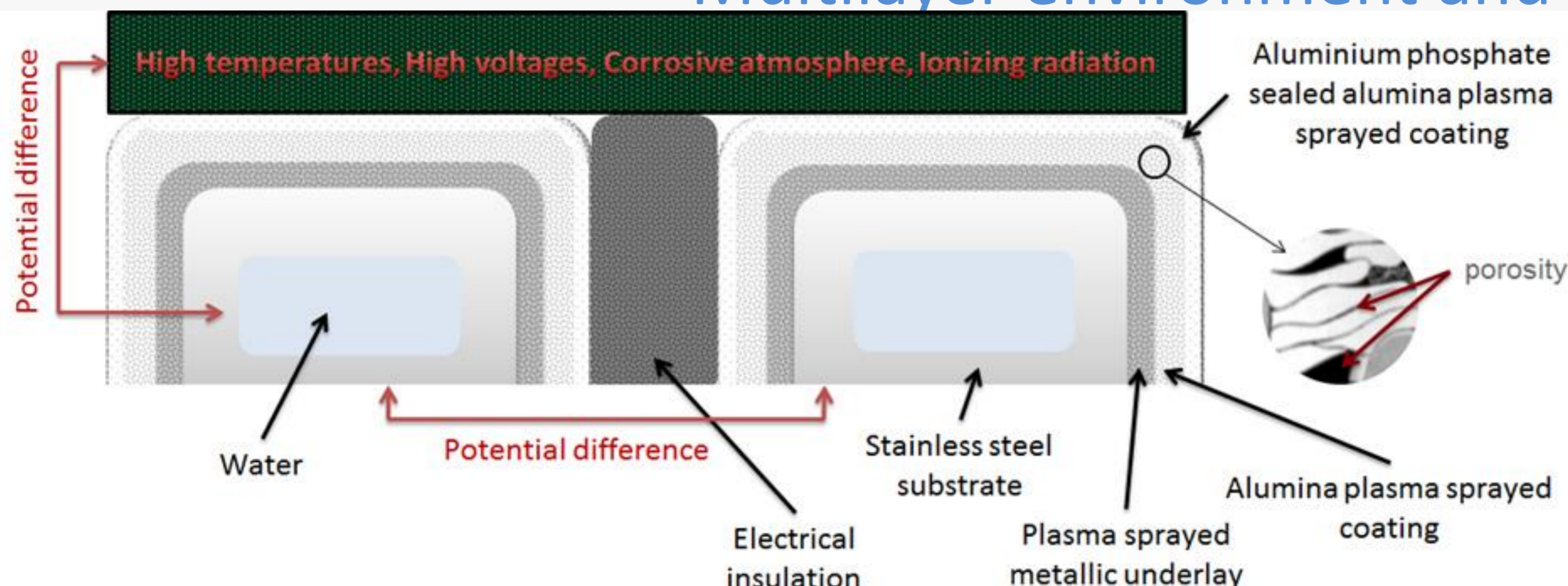
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Context - Objectives

To electrically protect stainless steel substrates over a wide area, the most commonly used technique is thermal spraying of alumina coatings. These coatings are known to form an electrical insulation barrier that protects efficiently the stainless steel structures subjected to widely varying voltages. However for specific applications, electrical conductor liquid or corrosive gas inlets require the open and/or interconnected porosity of the alumina coating to be sealed with aluminum phosphate. The sealing procedure was carried out by impregnation of the thermal sprayed alumina coating with a mono aluminum phosphate solution (MAIP) and applying heat treatment to the system. This procedure is characterized by parameters related to the impregnation technique (vacuum, brush and spray), the heat treatment and the impregnating solution. This study was intended to optimize the sealing procedure, based on an experimental design established from the factors impacting the sealing of the alumina coating deposited on a welded stainless steel.

Multilayer environment and composition



- Homogenization by mechanical stirring.
- Heat until 50°C-70°C.

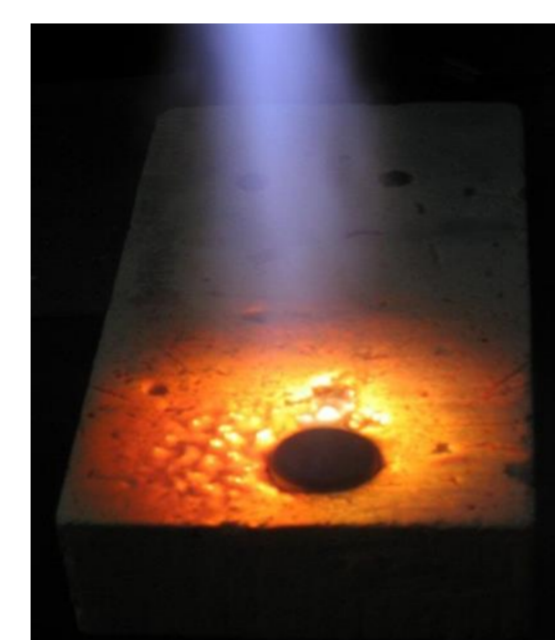


- Application onto the coatings.
- Heat treatment in the temperature range 400°C-500°C.

Characterization tests

Thermal shock tests

The samples were alternately heated by an oxyacetylene flame and cooled by a compressed air jet. The imposed heat flux was 0.6 MW.m⁻² to maximize the stresses supported in the industrial process. Samples were tested under 10 cycles from 250 to 450°C.



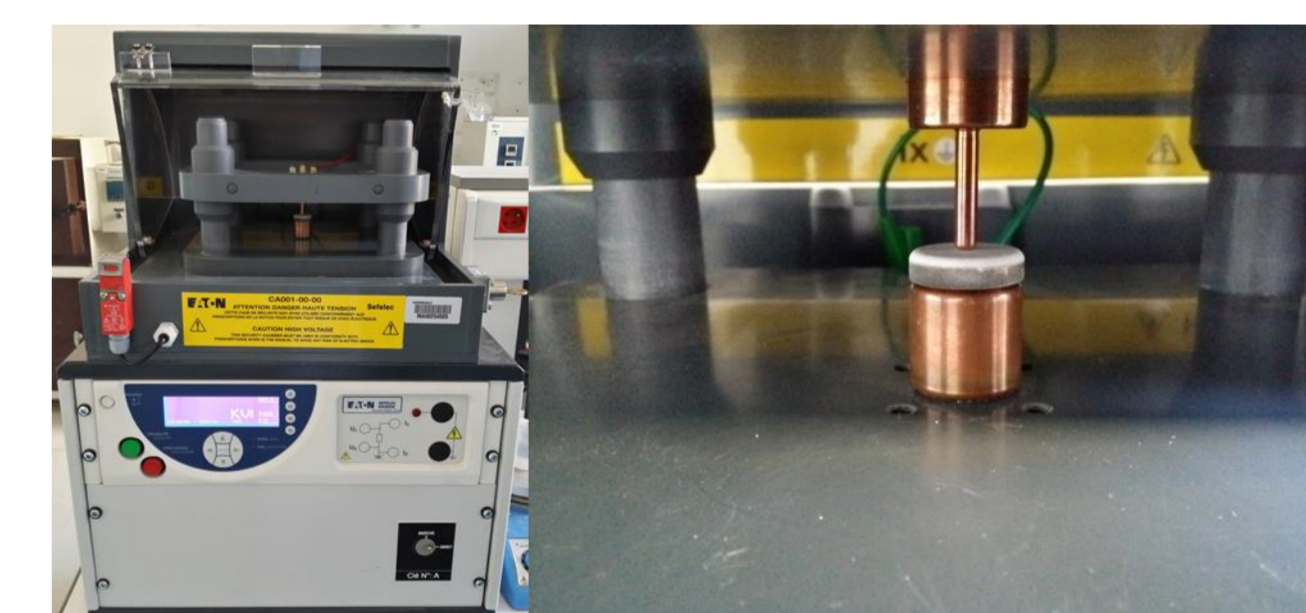
Thermal aging tests

The samples were placed in a furnace in air at a pre-set isothermal temperature. For this study, they were heated to 250°C. The duration of the thermal aging test was set at 100 hours.

Dielectric strength characterization (breakdown voltage)

The operator gradually increased the voltage from 0 to 2000 V in 30 seconds and observed the change in the electrical current. While the coating insulates, there was no current. The voltage which corresponded to the appearance of electrical current was breakdown voltage.

Measurements were carried out under both dry and wet conditions (the tip was placed in a droplet of a 70 g.L⁻¹ potassium chloride solution deposited on top of the coatings).



Results – Discussions

Studying the contributions of effects showed that both the impregnation technique and the impregnant have a major impact on the result for breakdown voltage. Therefore, the interactions of the heat treatment with the interaction of order two between impregnation technique and impregnant were analyzed.

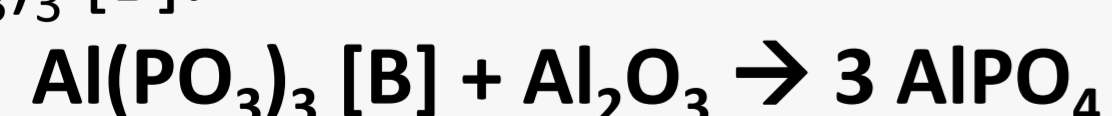
The experimental design established that the best aluminum phosphate sealing is obtained on samples prepared with **vacuum impregnation**, a **laboratory solution with a P/Al ratio = 3.5** and **with a long dwell time heat treatment in the temperature range 400-500°C**.

Experimental matrix

Factors	Levels	
Impregnation technique (TIM)	Brush	PIN
	Spray	PUL
	Vacuum	SVI
Heat treatment (TTH)	Without dwell time	PPA
	Short dwell time (one day) in the temperature range 400°C-500°C	P20
	Long dwell time (one week) in the temperature range 400°C-500°C	P50
Impregnating solution (IMP)	Budenheim P/Al = 3	BU3
	Budenheim P/Al = 3.5	B35
	Laboratory solution P/Al = 3	C3
	Laboratory solution P/Al = 3.5	C35
	Aged laboratory solution P/Al = 3	C3V
	Aged laboratory solution P/Al = 3.5	C5V

Nuclear magnetic resonance (NMR) showed two aluminophosphate phases formed during the sealing heat treatment [3]: **Al(PO₃)₃ [B]** and **AlPO₄**.

The presence of AlPO₄ can be attributed to the following reaction between alumina and Al(PO₃)₃ [B]:



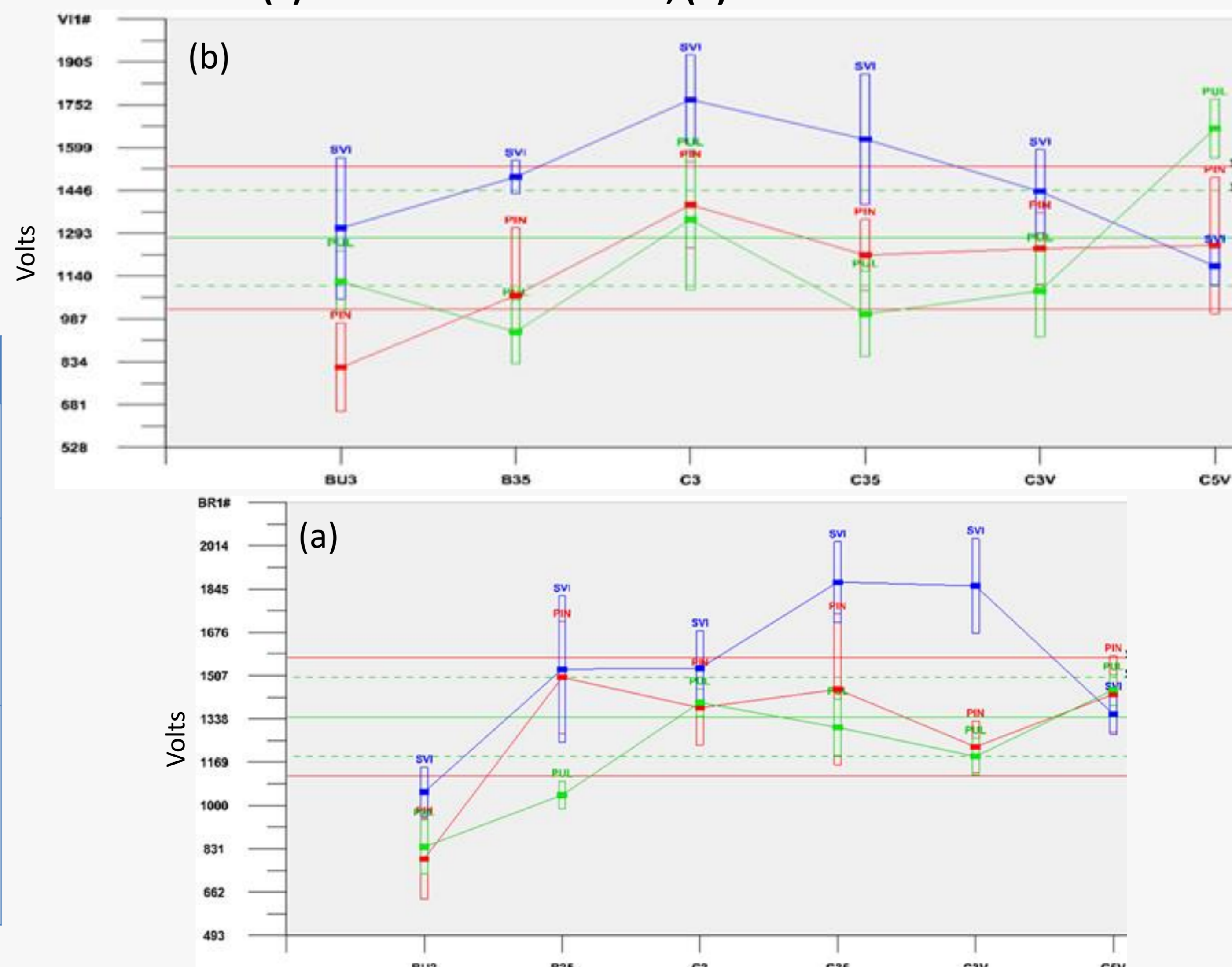
This reaction appears to be favored at low temperatures.

The more AlPO₄ present, the more efficient the coating.

Evolution of breakdown voltage (Volt)

Evolution of breakdown voltage (V) for samples impregnated with different impregnation techniques and solutions. These samples had a heat treatment with a long dwell time (P50):

(a) without thermal stress, (b) with thermal stress.



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 [3] G. Tricot, New insights into the thermal evolution of aluminophosphate solutions: a complementary XRD and solid state NMR study, Journal of European Ceramic Society, Vol 28, Issue 6, p1135-1141, 2008.