# Experiential learning - a comparison of the effectiveness of surface pre-treatment methods for surface active alloys

# Raimonda Lukauskaitė, Algirdas V. Valiulis & Olegas Černašėjus

Vilnius Gediminas Technical University Vilnius, Lithuania

ABSTRACT: Engineering graduates' generic skills in high degree can be developed only by experiential learning. The main opportunity for acquiring these skills is in the course work or final thesis. Coating components made of aluminium alloys causes serious problems. The bond strength of most aluminium alloy coatings is relatively weak because of a permanent, chemically continuous oxide barrier. Surface pre-treatment is one of the most important factors in achieving strong and stable adhesion. The most common treatments are mechanical abrasion, vapour degreasing and alkaline cleaning. Analysis of the surface of the cleaned specimens showed that different cleaning methods in high degree affect the surface of the aluminium alloy. This article focuses on work carried out by students during their Master's degree studies. This experiential learning project has proved successful in its aims, associated with practical skills and the use of modern technological equipment, and proved to be an effective motivator for research-based projects.

#### INTRODUCTION

Aluminium alloys have been considered to be one of the most useful and versatile materials because of their interesting mechanical properties, such as a high strength-to-weight ratio, high electrical conductivity and good thermal conductivity. They are also easy to shape, distinguish themselves because of their good corrosion resistance, are widely available and are relatively inexpensive. However, the low hardness and low melting point of such alloys results in poor tribological properties and heat resistance. Coating aluminium alloy components causes serious problems; namely, the bond strength of most coatings of aluminium alloys is relatively weak. One of the main reasons is because of rapid aluminium oxidation in the air. Under this condition, a permanent, chemically continuous oxide barrier is formed on the substrate. Al<sub>2</sub>O<sub>3</sub> has many desirable qualities, which include being chemically inert over a fairly broad range of pH, having high hardness and possessing certain barrier properties [1]. However, this oxide hampers a formation of a close contact between sprayed coating and the substrate. There are various substrate pre-treatment methods before coating, which eliminate oxide formation on aluminium alloys.

Surface pre-treatment is one of the most important factors that will allow strong and stable adhesion to be achieved. The function of this pre-treatment is to remove the unstable aluminium oxide/hydroxide film and to clean stubborn oil and grease off the bonding surfaces. There are numerous aluminium pre-treatment procedures, which are usually multi-stage procedures. The most common treatments are mechanical abrasion, vapour degreasing and alkaline cleaning. Mechanical treatment consists of a roughening process, such as by grit-blasting or scotch brite abrasion. The roughness increase frequently enhances adhesion. However, possible residual debris and mechanical damage to the adherent can be detrimental to bonding [2-3].

Grit blasting is widely used as pre-treatment, but this treatment is an environmentally unsound method because of scattered sludge and the noise created [4]. Vapour degreasing mainly consists of removing oil and other organic contaminants from the roughened surface. Alkaline degreasing consists of the immersion of the substrate in a suitable alkaline solution [2-3]. Chemical and electro- chemical pre-treatment of Al surfaces exhibit favourable adhesion. Disadvantages regarding those pre-treatment methods are the disposal of the chemicals involved, long pre-treatment times and high operating costs. For these reasons, the application of the mentioned or similar pre-treatment methods is not recommended in many industries. On the other hand, mechanical pre-treatments often result in unsatisfactory adhesion and long-term stability. The results of adhesion and long-term behaviour of excimer- and  $CO_2$ -laser treatments also show some restrictions [5]. The application of arc-discharge pre-treatment methods could be considered as an alternative technique. After removal of the oxide layers by arc-discharge, the surface is rough. The oxides under the cathode spots are vaporised and removed instantaneously. This technique of pre-treatment could be considered to fulfil both requirements: it is an environmentally friendly preparation method, and it could be developed to be fully automated in industry [4-5].

The aim of this article is to show how, during preparation of their course project or thesis, students can investigate the influence of the different cleaning methods on the parameters on surface cleaning area, roughness and surface free energy, also they can examine the microstructure, topography and chemical composition of aluminium alloys after the cleaning.

#### METHODS OF THE RESEARCH

The microstructure of the surface of aluminium specimens after surface cleaning can be examined by scanning electron microscope SEM SU 8000 with an SE detector of secondary electrons. The chemical composition of the surface of the specimen after cleaning can be analysed using x-ray microanalysis.

The roughness of the cleaned section of specimens can be measured by the use of roughness tester TR200. The topography of the cleaned aluminium surface can, then, be examined using 3D optical profilometer VEECO NT9300.

Surface free energy of aluminium alloys after their cleaning can be measured by the sessile drop method [6-8]. The sessile drop method is an optical measurement method based on contact angle, which can be used to evaluate solid surface wettability. Measurement of the contact angle can be carried out on a research stand. The stand consists of a picture channel (camera, feeder and connectors), a stereoscope microscope, a measurement table and an optical illuminator. The *Scion Image* program for computer image analysis can be used to measure the contact angles. The reagents used to test the surface free energy of aluminium specimens were distilled water/formamide and water/diiodomethane, which have high surface free energy and different surface free energy components, and they are insoluble in aluminium alloys.

## RESEARCH BY SCANNING ELECTRON MICROSCOPE

Analysis of the surface of the cleaned specimens through the use of a scanning electron microscope shows that the morphology of all specimens is different (Figure 1). During cathodic cleaning, students found that one or two cleaned zones appear, depending on the discharge current (Figure 1d). Analysing the surface of the cleaned aluminium specimens by use of a scanning electron microscope shows that cathodic cleaning can cause considerable changes to the surface microstructure - one zone with a partial destruction of the aluminium oxide film, and a second zone with total destruction of the aluminium oxide film.



Figure 1: The surface microstructure of aluminium specimens: a) non-cleaning specimen; b) specimen cleaning by 10% NaOH alkaline pre-treatment method; c) specimen cleaning by blasting pre-treatment method; and d) specimen cleaning by cathodic pre-treatment method.

X-ray microanalysis shows that after cathodic cleaning, in the zone where the oxide film  $Al_2O_3$  is fully destroyed, the mass concentration of oxygen is 4.775 weight %, while the mass concentration of oxygen on the surface of blasting cleaned aluminium specimen is 6.982 weight %.

The energy dispersive x-ray microanalysis of the alkaline cleaned specimen shows that the lowest quantity of oxygen 1.933 weight % is in Al alloy surface after using the 10% NaOH cleaning method, but non-cleaning Al sample quantity of oxygen is 8.590 weight %. This result shows that alkaline cleaning reduces the oxygen concentration on Al surface about 4.4 times.

### THE RESEARCH OF ROUGHNESS AND TOPOGRAPHY OF ALUMINIUM SPECIMENS

Roughness measurements show that the lowest roughness of the aluminium specimen occurs after 10% NaOH pretreatment and the highest roughness is after blasting with  $Al_2O_3$  of aluminium alloy (Figure 2). Increasing surface roughness of the substrate can influence a better adhesion of the coating.



Figure 2: 3D topography of surface of aluminium specimens, when: a) the non-cleaning specimen; b) the specimen is cleaned by alkaline pre-treatment method; c) the specimen is cleaned by blasting pre-treatment method; and d) the specimen is cleaned by cathodic pre-treatment method.

## THE RESEARCH OF SURFACE FREE ENERGY OF SPECIMENS BY THE SESSILE DROP METHOD

Analysis of the surface of the cleaned specimens by the sessile drop method shows that the surface free energy of all specimens is different (Figure 3).



Figure 3: The surface free energy of surface of aluminium specimens after pre-treatment methods.

The sessile drop method analysis shows the lowest surface free energy of the aluminium alloy occurs after cathodic cleaning at low cleaning current (40A). The highest surface free energy can be observed in the aluminium alloy after blasting with  $Al_2O_3$ . Higher surface free energy of the substrate affects the better wetting of liquid on the substrate surface.

The measurement of surface free energy after cathodic cleaning shows the influence of key parameters of cathodic cleaning (strength and frequency of current) upon changes of surface free energy (Figure 4).



Figure 4: The dependence of surface free energy of the cleaned aluminium alloy on the cathodic cleaning current strength and frequency.

## CONCLUSIONS

During the practical training and research elaborated here, students will be able to do a number of things. During experimental tests of cleaning of aluminium alloy specimens, students can investigate dependence of surface quality parameters (area of cleaned zone, cleaned surface roughness, surface free energy, etc) on the method of surface cleaning and its parameters (cathodic cleaning current strength, frequency and arc length or gap between the electrode and the cleaned substrate).

Analysis of the surface of the cleaned by cathodic cleaning specimens by a scanning electron microscope will lead to students finding that two different cleaning zones appeared on the surface of aluminium alloy. In the first zone of the aluminium specimen's surface, a partial destruction of the aluminium oxide film could be observed; in the second cleaning zone, total destruction of aluminium oxide film could be seen.

During research on surface free energy and using several reagents, students can compare the results and determine the accuracy of the sessile drop method.

Experiment-based study courses prove to be an excellent opportunity for those students with a strong interest in practical approach in engineering science and practice.

#### REFERENCES

- 1. Moffitt, C.E., Wieliczka, D.M. and Yasuda. H.K., An XPS study of the elemental enrichment on aluminium alloy surfaces from chemical cleaning. *Surface and Coatings Technol.*, 137, 188-196 (2001).
- 2. Prolongo, S.G. and Urena, A., Effect of surface pre-treatment on the adhesive strength of epoxy- aluminium joints. *Inter. J. of Adhesion & Adhesives*, 29, 23-31 (2009).
- 3. Joshi, S., Fahrenholtz, W.G. and O'Keefe, M.J., Effect of alkaline cleaning and activation on aluminium alloy 7075-T6. *Applied Surface Science*, 257, 1859-1863 (2011).
- 4. Hara, M., Yamakawa, S., Saito, M., Tobe, S. and Inaba, T., Bonding adhesive strength for plasma spray with cathode spots of low-pressure arc after grit blasting and removal of oxide layer on metal surface. *Progress in Organic Coatings*, 61, 205-210 (2008).
- 5. Anagreh, N. and Robaidi, A.A., Improvement in adhesion behavior of aluminium due to surfaces treatment with arc discharge. *Jordan J. of Mechanical and Industrial Engng.*, 4, **2**, 330-339 (2010).
- 6. Mazzola, L., Bemporad, E. and Carassiti, F., An easy way to measure surface free energy by drop shape analysis. *Measurement*, 45, 317-324 (2012).
- 7. Rudawska, A. and Jacniacka, E., Analysis for determining surface free energy uncertainty by the Owen-Wendt method. *Inter. J. of Adhesion & Adhesives*, 29, 451-457 (2009).
- 8. Tan, Y. and Guo, M., Using surface free energy method to study the cohesion and adhesion of asphalt mastic. *Construction and Building Materials*, 47, 254-260 (2013).