

Time dependent argon glow discharge treatment of Al-alloy samples

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Abstract. Aluminium alloy ultra-high vacuum system provides a convenient tool to access the UHV region due to short pump down time, its reduced weight, low cost etc. For UHV systems, aluminium and its alloys are preferred materials to stainless steel.

A cylindrical discharge chamber of SS 304 with various ports on it, evacuated by turbomolecular pumping unit is used in the experimental system. A hollow cathode dc glow discharge in argon for different time durations is used to treat chemically cleaned ASA 6063 aluminium alloy samples, keeping all other parameters constant.

The scanning electron microscope (SEM) is used to examine processed surfaces and to study topographical features. The energy dispersive microanalysis by X-rays (EDX) is used to determine the elemental composition of the samples.

The results indicate the physical sputtering taking place in Ar GDC. The etched area increases with discharge time duration. The EDX spectrum shows the inconsistency in weight percentage of various elements of Al-alloy.

Keywords. Aluminium alloy; scanning electron microscope; energy dispersive microanalysis by X-rays; argon glow discharge; time-dependent.

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1. Introduction

Stainless steel and aluminium alloy are the most commonly used materials for vacuum chambers [1] as well as storage rings [2]. Comparative study of outgassing rates show that stainless steel can be replaced by aluminium alloy for UHV chambers [3]. The short pumpdown time, great reduction in bakeout temperature and time together with its reduced weight and low cost, the aluminium alloy ultra high vacuum system provides a remarkably convenient tool to access the UHV region [4]. There is not much information available on the effects of GDC on the surface structure of aluminium and its alloys. This paper discusses the effect of argon GDC on ASA 6063 aluminium alloy surfaces.

2. Motivation

The study of change in surface properties of metals and their alloys under plasma processing has a wide range of applications. All the applications of non-equilibrium plasmas involve processes like sputtering, etching, polymerization, surface modifications, etc. The glow discharge conditioning (GDC) has become the final and essential stage of surface conditioning process of vacuum vessels of particle accelerators, storage rings and tokamaks [5]. GDC has the advantage that it is very simple to apply. Also it can be used for surface preparation of vacuum components and vacuum system of any size. This forms the main motivation to carry out the work described in this paper.

3. Theoretical background

SEM: Scanning electron microscope is widely used to examine surfaces and study topographical features [6]. In SEM, resolution down to a few thousand angstroms is possible, depending on nature of the sample. A focussed electron beam scans the surface and the intensity of secondary electrons is monitored. The output from the secondary electron detector can be shown on a cathode ray tube, and the strength of the image at each point varies according to the intensity of secondary electron production.

EDX: The energy dispersive microanalysis by X-rays [7] is carried out to determine the elemental composition of the sample. In the energy dispersive analysis the emitted x-rays of all wavelengths are collected and are sorted electronically by the system.

In this work, samples of aluminium alloy are treated under dc glow discharge conditioning for different time durations, keeping all other parameters constant. Untreated and treated samples are analysed using SEM micrographs and spectra obtained from EDX.

4. Apparatus and experimental procedure

The experimental system used in the study is as shown in figure 1. The system consists of two vacuum chambers connected by manually operated gate valve. The main discharge chamber is SS304 cylinder having inner diameter 10 cm and length 50 cm with various ports on it. The surface area of discharge chamber is 2100 cm² having total volume of 4.5 litre. Another chamber of same diameter and height 15 cm has a quadrupole mass analyser mounted on it for residual gas analysis. The entire system is evacuated by a turbomolecular pumping unit (TMP) with a turbomolecular pump (50 l s⁻¹), a two stage rotary pump (1.52 m³ h⁻¹) and a vent control unit. To measure the low pressure, a Bayard Alpert (BA) ionization gauge (IG) is used. In the discharge chamber, where pressure is of high order in the range 10⁻¹ to 10⁻³ mbar, a convection gauge (CG) is used. For a hollow cathode glow discharge 5 kV, 100 mA power supply is used. The anode is a small SS plate while the grounded body of the chamber acts as cathode.

The glow discharge schedule for the experiments is as follows:

- (a) The vacuum system was evacuated to a base pressure of 1×10^{-6} mb, then baked and naturally cooled, so that a steady state pressure of 3×10^{-7} mb was reached.

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- (b) The gas inlet system was also simultaneously evacuated to a pressure below 2×10^{-2} mb and the process gas was filled in it.
- (c) The process gas argon was introduced in the system via a leak valve so that the pressure in the system was about 1×10^{-4} mb.
- (d) The discharge chamber was throttled such that the pressure in the discharge chamber was 4×10^{-2} mb.
- (e) When equilibrium conditions were reached, the discharge was struck. The discharge current was adjusted to obtain the required dc current density.
- (f) The discharge was terminated after continuing it for a desired period.
- (g) The leak valve was closed and the system was evacuated to a stable pressure.

The plasma parameters found by single probe method were: electron temperature = 2.0 eV and ion density = 5×10^9 per c.c. for fixed value of current density $20 \mu\text{A}/\text{cm}^2$ and discharge pressure 4×10^{-2} mbar.

Chemically cleaned aluminium alloy samples of size $1 \text{ cm} \times 1 \text{ cm} \times 0.1 \text{ cm}$ were used. The glow discharge treatments were performed at three different discharge durations $t_D = 1, 2$ and 3 hours. The other parameters viz. current density $J_D = 20 \mu\text{A}/\text{cm}^2$, wall temperature $T_D = 25^\circ\text{C}$ and the discharge pressure $P_D = 4 \times 10^{-2}$ mbar were the same in all the treatments. The untreated and plasma processed samples were analysed using SEM and EDX.

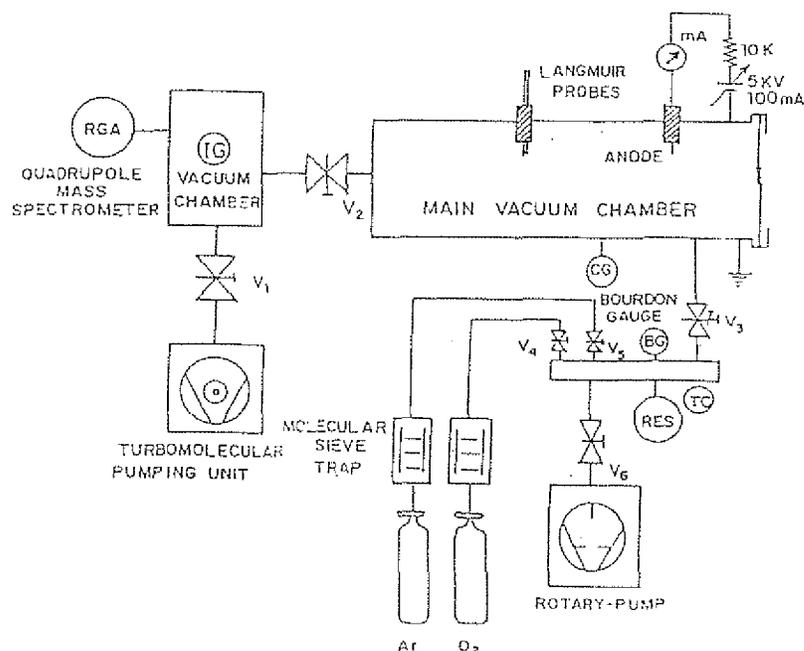


Figure 1. Schematic representation of the experimental system.

5. Results and conclusion

The results of SEM micrographs (figures 2 to 4) indicate that the bombarding ions erode the surface and pits are formed due to etching of the surface by ions. The etching is observed at more places on the sample surface and etched area increases as discharge time duration increases.

As the magnification of SEM micrograph increases, the area under observation gets reduced and therefore entire area of the lower magnification micrograph is not covered. Hence it is difficult to do the quantitative analysis.

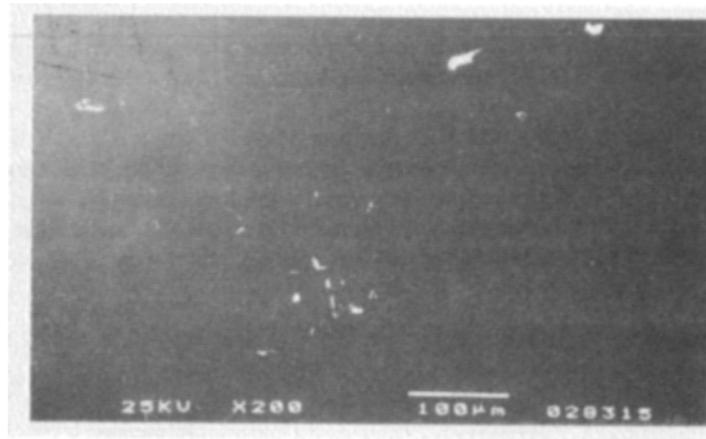


Figure 2. SEM micrograph of the ASA 6063 Al-alloy sample after Ar GDC treatment. $t_D = 2$ hours, $J_D = 20 \mu A/cm^2$, $P_D = 4 \times 10^{-2}$ mbar.

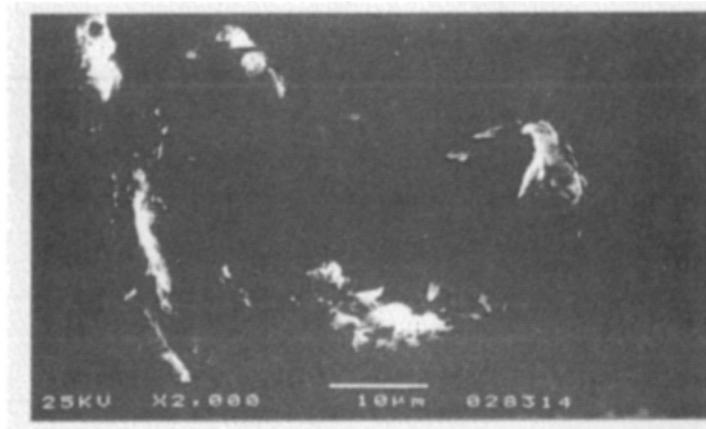


Figure 3. Higher magnification SEM micrograph of some of the structures shown in figure 2. $t_D = 2$ hours.

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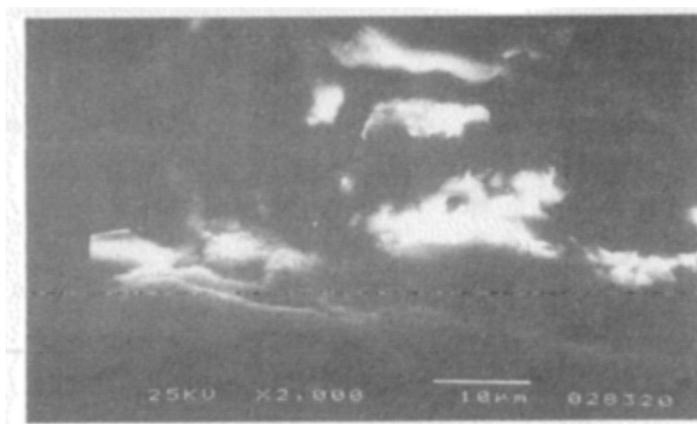


Figure 4. SEM micrograph of the Al alloy ASA 6063 sample after Ar GDC treatment. $t_D = 3$ hours, $J_D = 20 \mu A/cm^2$, $P_D = 4 \times 10^{-2}$ mbar.

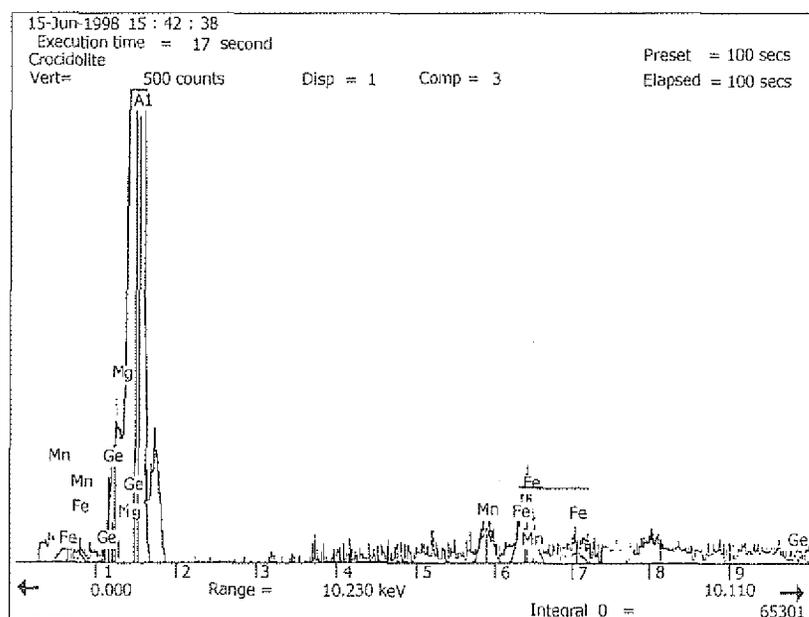


Figure 5. EDX spectrum of treated sample showing the elements present.

The EDX spectrum (figure 5) shows the peaks corresponding to elements aluminium, magnesium, iron, germanium and manganese. The absence of any other peak in untreated and treated samples indicates that no contamination of sample surface has taken place.

The results of EDX analysis showing weight percentage of elements is given in the following table. One of the EDX spectra is shown in figure 6.

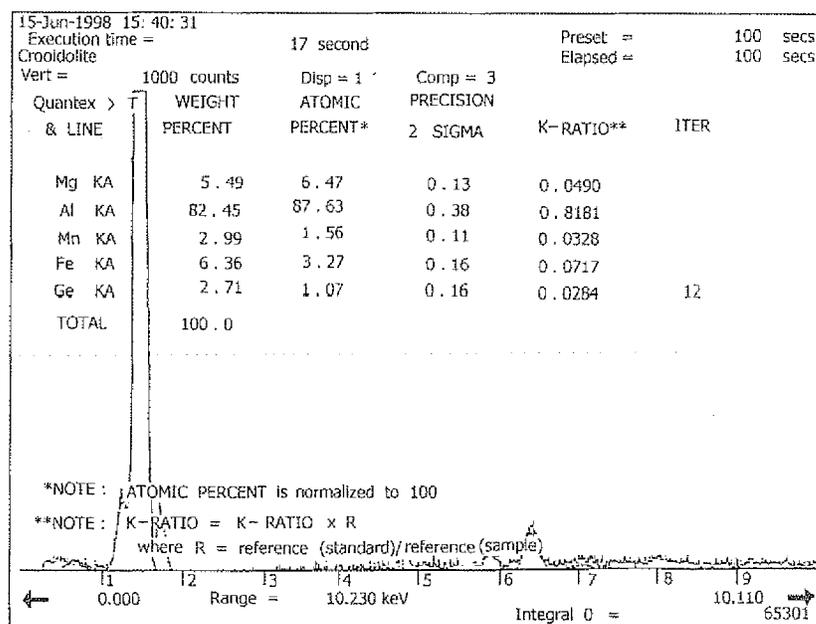


Figure 6. EDX spectrum of treated sample. $t_D = 1$ hour.

Table. EDX analysis showing weight percentage of elements.

Element	Discharge time duration			
	0 hour	1 hour	2 hours	3 hours
Magnesium (Mg)	5.48	5.49	4.74	4.48
Aluminium (Al)	85.74	82.45	83.90	83.26
Manganese (Mn)	1.93	2.99	3.03	3.40
Iron (Fe)	3.75	6.36	5.06	5.74
Germanium (Ge)	3.10	2.71	3.28	3.12

From the above table for weight percentage of elements in EDX analysis it is observed that:

- (i) For magnesium, relative weight percentage is almost same for 1 hour discharge duration. Then, it decreases with increase in time duration.
- (ii) For aluminium, iron and germanium, relative weight percentage decreases or increases inconsistently with time of discharge duration.
- (iii) Relative weight percentage of manganese continuously increases with discharge time duration.
- (iv) With respect to untreated sample, for 1 hour discharge duration, the relative weight percentage of manganese and iron increases, while, for Al and Ge it decreases. For Mg, it practically remains the same.

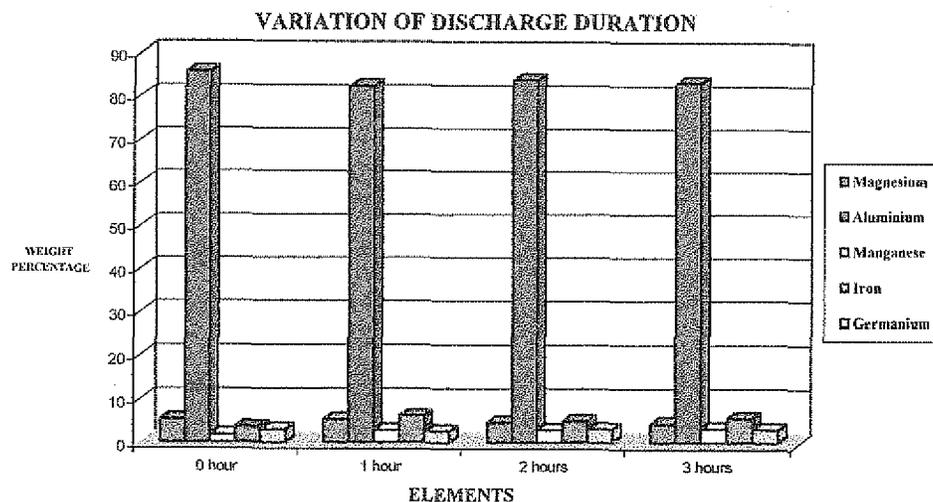


Figure 7. Bar graph showing weight percentage of various elements for different discharge duration.

- (v) For 2 hours and 3 hours discharge duration the relative weight percentage of Mg and Al decreases, while for Mn, Fe and Ge it increases with respect to untreated sample.
- (vi) For 1 hour discharge duration the relative weight percentage of Al and Ge is minimum and that of Fe is maximum compared to other time durations.

There is inconsistent increase or decrease in the weight percentage of various elements of Al alloy. This is because of the non uniform etching over the entire surface of the sample due to random motion of ions in the discharge, that are bombarding the sample surface.

The plasma parameters are found to be electron temperature = 2.0 eV and ion density = 5×10^9 per c.c.

The bar graph showing weight percentage of various elements for different discharge durations is as shown in figure 7.

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