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# Surface modification of aluminum using ion nitriding and barrel nitriding

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**Abstract**

In this study, an aluminum nitride (AlN) layer was formed on the surface of aluminum in barrel with alumina/aluminum–magnesium alloy powder, and gas and ion nitriding was thus enabled. The influence of process parameters on the film thickness, hardness and constituents of AlN layer was examined. The film thickness was compared for three nitriding methods: firstly, barrel nitriding only; secondly, gas nitriding after barrel nitriding; and thirdly, ion nitriding after barrel nitriding. After nitriding, there are two layers on the substrate, one is the modified layer composed of AlN and the other is a deposited overlayer composed of alumina and AlN. With increase in time in the barrel, the thicknesses of the modified layer and deposited overlayer also increase. In the combined processes, the thickness of the modified layer is greater than that obtained by barrel nitriding alone. In addition, it is possible that the thickness of the deposited overlayer decreases when gas or ion nitriding is used. While hardness of the modified layer combined with gas or ion nitriding is increased, that of the modified layer combined with ion nitriding is the highest.


*Keywords:* Barrel nitriding; Ion nitriding; Aluminum alloy; Aluminum nitride; Gas nitriding; Aluminum–magnesium alloy powder

## 1. Introduction

The use of aluminum (Al) or non-metallic materials, such as resin, instead of steel for the lightening of car components or machine parts has recently increased. Al and its alloys have advantages over non-metallic materials: aluminum alloys have a high melting point, a good corrosion-resistant, a good workability and have a good thermal conductivity. However, the hardness and wear resistance of Al alloys are respectively lower and inferior to those of steel; therefore, there is a limit in their application to sliding parts. Hence, research has been carried out in surface modification technology to increase the applicability of Al alloys as sliding parts. Surface modification technologies for Al alloys can be classified into three main groups. Alloying is the first method and this forms a hard film on the Al surface [1–3]. The second group is coating method, which covers the Al surface with hard materials [4–6]. The third is a heat treating process, such as ion or gas nitriding [7,8]. Generally, in the aluminum anodizing, which is one of the alloying methods, the modified layer's hardness is moderate, but in the surface melting process for alloying by heating, a thick and hard film can be produced [9,10]. Coating methods include ion plating [11], composite plating [12], thermal spraying [13,14], cladding, etc. In general, these methods can produce a very hard film, but the film growth rate is low and it is difficult to form thick films, except for thermal spraying. Moreover, in most of the surface modification methods, adhesion to the Al substrate is low. Recently, an experiment has been carried out with gas or ion nitriding of Al substrate, with the aim to obtain a hard aluminum nitride (AlN) layer. However, the formation of a nitride layer by nitrogen diffusion is difficult, because the oxide film that exists on the Al surface is very dense. Furthermore, the formation of Al oxide occurs more readily than AlN, because of better affinity between Al and oxygen in comparison with nitrogen. Therefore, oxidation by oxygen or water vapor present in the atmosphere occurs more than nitriding can. To create AlN below the melting point temperature is difficult, because nitriding is inhibited by oxidation reactions. Therefore, equipment that removes

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the oxygen and activates the nitrogen in the atmosphere is necessary in order to enable nitriding of the Al surface. The author used a fluidized bed to solve these problems and succeeded in the nitriding of Al [8]. However, a new problem appeared, that is a lot of nitrogen was necessary in fluidized bed for nitriding to occur. A barrel was used to solve this new problem. The barrel was added with alumina ($Al_2O_3$) powder and an aluminum–magnesium (Al–Mg) alloy powder; these powders flow like a liquid when the barrel is swung. Barrels are generally used in various fields, such as plating and polishing. In this study, an AlN layer was formed on the surface of Al substrate in the barrel and, subsequently, gas nitriding or ion nitriding was carried out. The thickness and hardness of AlN layer were examined as a function of the process parameters.

## 2. Experimental procedure

The substrate used was 10 width×20 length×5 thickness (mm) of JIS-A1050 commercial grade pure Al. $Al_2O_3$ particles (average diameter 0.1 mm) and Al 50 wt.% Mg powder (average diameter 0.2 mm) were used as filler of the barrel. A schematic diagram of the barrel and gas or ion nitriding furnace used for the experiment is shown in Fig. 1. The barrel was added with the 1.0 wt.% Al–Mg/$Al_2O_3$ powder and the specimen was set. The furnace temperature was adjusted with a temperature control unit. Nitrogen gas ($N_2$) was introduced into the barrel; the $N_2$ flow rate was adjusted with a program control unit and a mass flow controller. The furnace was evacuated by a rotary pump and the atmosphere was substituted by $N_2$. After the $N_2$ introduction, the specimen was heated to a fixed temperature and barrel nitriding was started. After a fixed time, the specimen was transferred from the Al–Mg/$Al_2O_3$ powder to the upper part of the barrel, in gas nitriding or ion nitriding section. In ion nitriding, the inside of the barrel was evacuated to a fixed pressure and DC voltage was applied; a glow discharge was generated between the chamber and the specimen, and ion nitriding was carried out. After a fixed time, the DC voltage was switched off and the specimen was cooled in the barrel. The temperature inside of the powder was measured by the thermocouple; the temperature, and $N_2$ and argon (Ar) gas flow-rate were monitored with a data processor. The basic heating and flow-rate profile of the nitriding process are shown in Fig. 2. The barrel and ion nitriding process parameters are shown in Table 1. Optical microscopy was used for thickness measurements and structure observations of the modified layers. X-ray diffraction analysis was carried out to characterize the constituents of the modified layers. The distribution of N, O, Al and Mg in the modified layers was analyzed by EPMA.

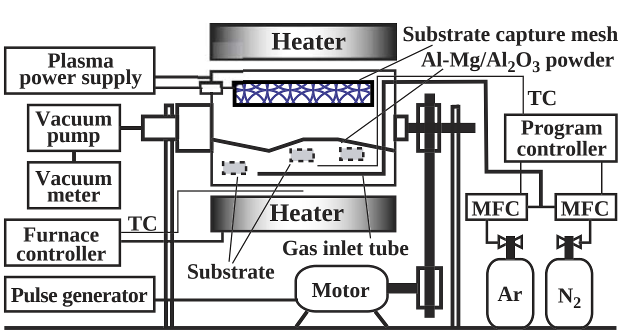


Fig. 1. Schematic diagram of barrel and gas or ion nitriding system.

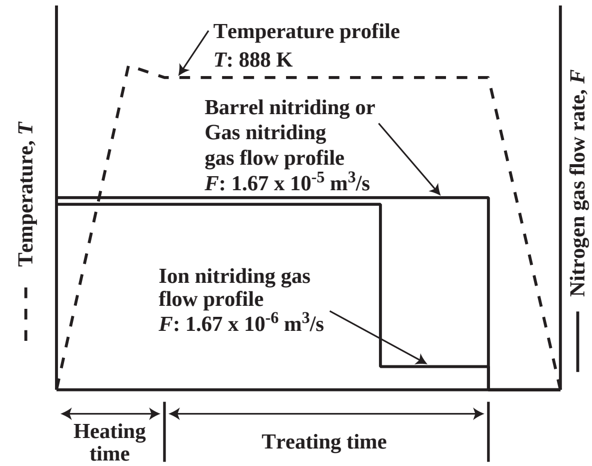


Fig. 2. Typical heating and gas flow rate profiles of the barrel and combined nitriding.

## 3. Results and discussion

### 3.1. Effect of the process parameter on modified layer formation and removal of deposited overlayer

When the $N_2$ flow rate during heating time was 100 $cm^3$/min, barrel nitriding failed. Whereas, when the flow rate is 1000 $cm^3$/min, a modified layer is formed on the Al substrate. Furthermore, if the small vessel made by stainless

Table 1
Treatment conditions

| | |
|---|---|
| *Barrel nitriding or gas nitriding* | |
| Nitriding temperature $T$ (K) | 888 |
| Heating time (ks) | 5.4 |
| Nitriding time $t$ (ks) | 10.8–18.0 |
| Nitriding gas | Nitrogen |
| Nitrogen gas flow $F$ ($10^{-6}$ $m^3$/s) | |
| During heating | 1.67 or 16.7 |
| During nitriding | 16.7 |
| Filler | $Al_2O_3$ (average diameter 0.1 mm) Al–50 wt.% Mg (average diameter 0.2 mm) |
| Al–Mg/$Al_2O_3$ ratio (wt.%) | 1.0 |
| *Ion nitriding* | |
| Nitrogen gas flow $F$ ($10^{-6}$ $m^3$/s) | 1.67 |
| Pressure (Pa) | 50 |
| Plasma voltage $E$ (V) | 500 |
| Plasma current $I$ (mA) | 100 |