

GROWTH OF THE AlN AND Fe-Al INTERMETALLIC COMPOUND MULTILAYER AS DIFFERENT NICKEL CONCENTRATION

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Keywords: Barrel nitriding, Aluminum nitride (AlN), Intermetallic compound (Fe₂Al_{5,4}), Multilayer, Bonding

Abstract

A great deal of research is being carried out on forming a multilayer on the material instead of single layer. However, most studies of multilayer formation have been conducted using the plasma processing or spray coating method only for surface modification. Recently, a new nitriding process has been proposed to produce an aluminum nitride on the aluminum surface by using a barrel. In addition, this study proposes a new concept on the production of a multilayer, which has an AlN and Fe-Al intermetallic compound layer, between aluminum and stainless steel by using the barrel nitriding. By forming a multilayer at the interface between the different substrates, this process can be applied to as a bonding process. The barrel nitriding was carried out at 640°C for 10hrs with Al₂O₃ powder and Al-Mg alloy powder. After barrel nitriding, the aluminum nitride (AlN) layer and Fe-Al intermetallic compound (Fe₂Al_{5,4}) layer were formed at the interface between pure aluminum and stainless steel. In addition, the thickness of the multilayer decreased with increasing the Ni concentration of stainless steel.

1. Introduction

In recent years, the automotive and aerospace industries have increasingly employed aluminum and aluminum alloys in manufacturing due to their light weight, high specific strength, good machinability, formability and corrosion resistance compared to most steels. However, they lack good wear resistance. The wear resistance of aluminum or aluminum alloys can be improved by surface hardening processes, such as nitriding treatment. The formation of aluminum nitride on the surface of Al alloys is one of the better known surfaces hardening processes with desirable properties which have high thermal conductivity (320 W/m K), good corrosion resistance, high electrical resistivity, high hardness value and high wear resistance [1-4]. In recent years, nitriding method using a barrel which has an effect of the physical-polishing for forming a nitride layer after remove a natural oxide film of an aluminum has been reported [5]. In addition, multilayer of AlN and FeAl could be formed at the interface between the aluminum and steel after barrel nitriding. As known well, the Fe-Al intermetallic compounds are regarded as promising materials for industrial applications because of their high specific strength, high hardness value, high temperature strength, as well as excellent oxidation and corrosion resistance except the poor workability [6-9]. The combination of these two layers at the interface between aluminum and steel, it can be lead to the high bonding strength without degradation of their properties. Most of bonding method is being preceded by brazing or welding method [10-11]. However, these

methods were accompanied by the degradation of their properties in the melting zone. Therefore, it is expected that this new method by using barrel nitriding can be applied to the bonding process on the aluminum and steel without degradation of substrates.

In addition, there is no report on the formation and growth rate of the multilayer which is composed of the AlN and Fe-Al compound under the condition of different alloying element. In this study, the formation and growth of the multilayer which has an aluminum nitride (AlN) and Fe-Al intermetallic compound multilayer as different nickel concentration of steel was investigated by using aluminum and various stainless steel.

2. Experimental Procedures

2.1 Specimen preparation

JIS-A1050 commercial grade pure aluminum was used as a substrate material, with a size of 20 mm width × 50 mm length × 3 mm thickness mm. In addition, SUS430 ferritic stainless steel, SUS304 and SUS316L austenitic stainless steels were prepared and machined to the same size as the pure Al for forming the intermetallic compound layer. The specific chemical compositions measured by a spectrometer (Ametek., Spectrolab) and the results are shown in Table 1.

Table 1 Chemical composition of A1050 and SUS304.

Specimen	Si	Fe	Cu	Mn	Mg	Zn	Ti	Al
A1050	0.0951	0.318	0.014	0.0028	0.0041	0.0038	0.0138	Bal.
Specimen	C	Si	Mn	P	S	Ni	Cr	Fe
SUS430	0.0364	0.268	0.631	0.0353	0.0171	0.288	15.61	Ba.
SUS304	0.0395	0.515	0.908	0.0172	0.0041	7.240	19.20	Bal.
SUS316L	0.0300	1.000	2.000	0.0450	0.0300	15.000	16.000	Bal. Mo(2.00)

2.2 Barrel nitriding

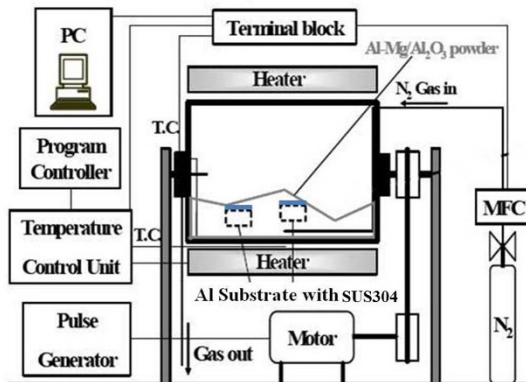


Fig. 1 A schematic diagram of the barrel nitriding process.

Al₂O₃ particles (average diameter 0.1 mm) and Al 50 wt.% Mg alloy powder (average diameter 0.2 mm) were prepared as filler for the inside of the barrel chamber. The furnace was evacuated by a rotary pump and the vacuum rate was maintained at 7.5×10^{-1} torr before the treatment; after that, the atmosphere was substituted by N₂. The N₂ gas flow rate was adjusted with a program control unit and a mass flow controller when the nitrogen gas (N₂) was introduced into the barrel. The furnace temperature was adjusted with a temperature control unit. A schematic diagram of the barrel nitriding furnace used for the experiment is shown in Fig. 1.

After the introduction of N₂, barrel nitriding was carried out with the oscillation of the barrel chamber at the temperature of 640 °C for 10hrs. After a holding time, the specimen was cooled in the barrel. The temperature inside of the powder was measured by a thermocouple; the temperature and the N₂ gas flow-rate were both monitored with a data processor.

2.3 Microstructure observation

The optical microscopy (OM) and scanning electron microscopy (SEM-EDS) (HITACHI, SU6600) were used to observe the micro-structure and morphology after grinding and polishing on the cross-sectional region of the specimen. The structures of the AlN and Fe-Al intermetallic compound layers were identified by an X-ray diffractometer (RIGAKU, UltraX 18 TTR) using monochromatic Cu-K_α radiation. The X-ray diffraction (XRD) measurements were performed using a goniometer at a scanning range of $30^\circ \leq 2\theta \leq 80^\circ$ at 40kV and 200mA with a step-size of 0.02°. The Al substrate was mechanically polished to the AlN and Fe-Al intermetallic compound layer in regular increments in order to measure the XRD for each layer. In addition, to investigate the behavior of the element for Ni, Mn and Si, EPMA (JEOL, JXA-733) analysis was performed.

2.4 Measurement of hardness

The micro hardness of the AlN and Fe-Al intermetallic compound layer was measured by using a Vickers micro hardness tester (Akashi, HM-125). The hardness measurements were performed from the Al substrate to the SUS304 stainless steel with the AlN and Fe-Al intermetallic compound layer under a load of 100 g for 10 sec on the cross-sectional region.

3. Results and Discussions

Fig. 1 shows the SEM micrograph and the EDS analysis results on the multilayer after the barrel nitriding at 640 °C for 10hrs. The barrel process performed with the A1050 pure aluminum and the SUS430 ferritic stainless steel after fixing.

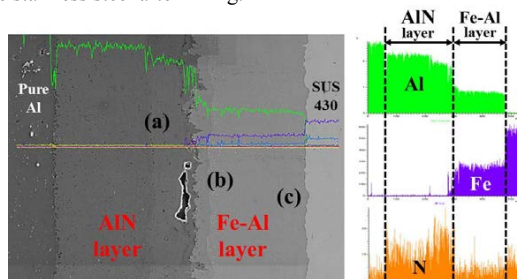


Fig. 1 SEM micrograph and the EDS line profile of Al, Fe and N on the aluminum nitride and Fe-Al intermetallic compound multilayer after barrel nitriding at 640 °C for 10hrs.

As shown in SEM micrograph, two kinds of layers were observed at the interface between the pure aluminum and ferritic stainless steel. To investigate the elementary composition the EDS analysis was carried out. From the EDS results, it is believed that the nitride layer and Fe-Al intermetallic compound layer were formed at the interface between pure Al and SUS430 ferritic stainless steel. As shown in the EDS profile, a large amount of Al and Fe is detected with nitrogen at each layer. The quantity of Al is gradually decreased as it closed to the Fe_xAl_y layer from the boundary of the aluminum substrate and AlN layer. After that, it is rapidly decreased on the Fe_xAl_y intermetallic compound layer and gradually decreased as approached to the SUS430 stainless steel. In addition, in contrast to the behavior of the Al, the Fe concentration is increased as it closed to the SUS430 substrate from the boundary of the AlN and Fe_xAl_y layer. And where it is considered to be the nitride layer in the next pure aluminum, a large amount of nitrogen was detected.

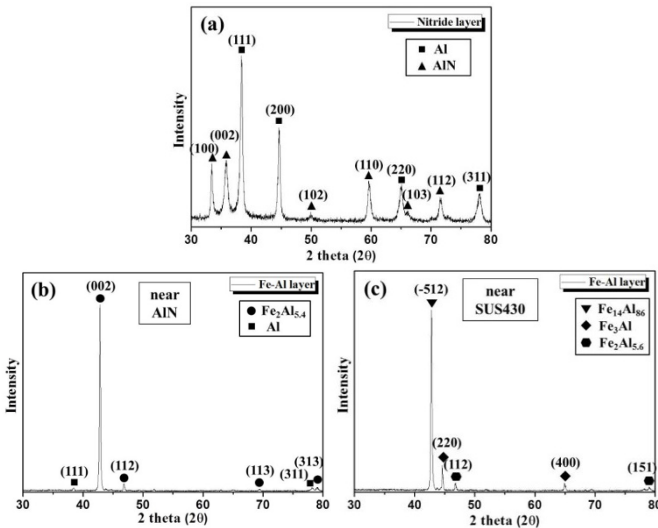


Fig. 2 X-ray diffraction patterns of the (a) AlN layer, (b) Fe-Al (near AlN) and (c) Fe-Al (near SUS 430 substrate) intermetallic compound layer between the interface of pure Al and SUS430 after barrel nitriding at 640 °C for 10hrs.

To clearly demonstrate the phase of the multilayer after barrel nitriding process, the XRD analysis was carried out on the layer of (a), (b) and (c) of Fig. 1. On the layer of (a) where it is considered to be the nitride layer, the Al and AlN peaks were detected. At the region of (b) where is near AlN layer, the $Fe_2Al_{5.4}$ and Al peaks were detected. In addition, the region of (c) where it is next to the SUS430 substrate shows the Fe_3Al , $Fe_{14}Al_{86}$ and $Fe_2Al_{5.6}$ intermetallic compound peaks. Both of the regions of intermetallic compound layer, most of peak indicate the Al rich compound. Whereas, the Fe_3Al peaks of the region (c) means the Fe rich compound. That is, it means that the Al was diffused from the pure aluminum substrate to the SUS430 substrate.

As it known well, the SUS430 is the ferritic stainless steel which has no Nickel element. However, SUS304 and SUS316L austenitic stainless steels contain a large amount of the Ni. In addition, the structure is changed to the FCC due to the addition of the Ni which is austenitic

stable element on the steel. To investigate of the effect of Ni for growth of the multilayer, the SUS304 and SUS316L austenitic stainless steel was used with A1050 pure aluminum.

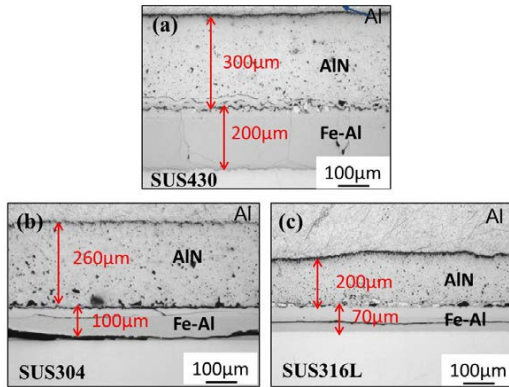


Fig. 3 Optical micrographs of the multilayer which has AlN and Fe-Al, between the pure Al and stainless steels, (a) SUS430, (b) SUS304, (c) SUS316L.

Fig. 3 shows the optical micrographs of multilayers which are composed of AlN and Fe-Al between the aluminum and various stainless steel after barrel nitriding 640 °C for 10hrs. As shown in the Figures, the thickness of multilayer was decreased with increasing the Ni concentration of stainless steel. In addition, the result from the XRD results as shown in Fig. 4, the AlNi₃ peaks were detected at the Fe-Al intermetallic layer of the SUS304 and SUS316L austenitic stainless steels. However, as mentioned above it, the Ni compound was not detected on the intermetallic compound of SUS430 ferritic stainless steel. That is, the Al was reacted with the Ni of the austenitic stainless steel when the Al diffused from the surface to interior of the stainless steel. The total thickness of the multilayer showed about 500 μm, 360 μm and 270 μm at the SUS430, SUS304 and SUS316L, respectively. In particular, the ferritic stainless steel has a thicker multilayer at the interface with the pure Al compare with the austenitic stainless steels. As it known well, the atomic packing factor of BCC (ferritic phase) and FCC (austenitic phase) is 68% and 74% respectively. That is, it means that the diffusion of Al is easier at the BCC ferrite phase due to the low packing density than the FCC austenite phase.

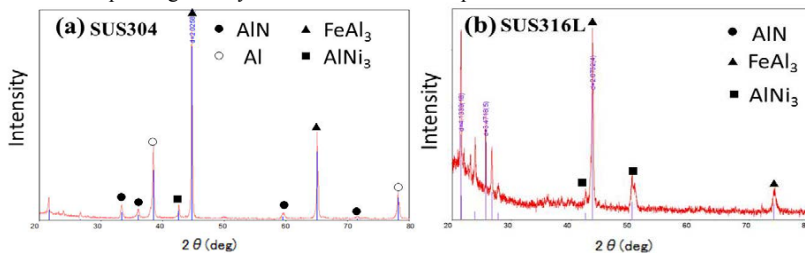


Fig. 4 XRD analysis results on the Fe-Al intermetallic layer of the (a) SUS304 and (b) SUS316L stainless steel.

To investigate the hardness of multilayer, the hardness test was performed. As shown in Fig. 5, the hardness of the ferritic and austenitic stainless steel shows around 200 HV. And the hardness of A1050 pure aluminum substrate showed about 30 HV. However, at the layer of the Fe-Al intermetallic compound, the hardness remarkably increased up to about 900 HV. The high hardness value on the Fe-Al intermetallic compound showed same tendency although the thickness of compound layer is different at various stainless steel. In addition, the AlN layer which was formed by the permeation of nitrogen atoms shows about 270 HV. From the results of the hardness test, the significant changes in hardness were not found as different Ni concentration, except for the thickness.

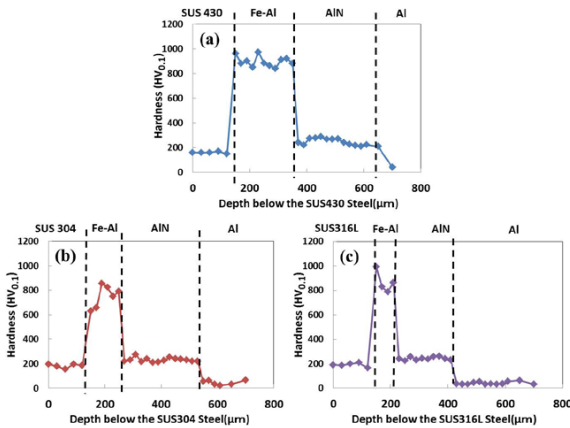


Fig. 5 Hardness variation of the Fe-Al and AlN multilayer as different stainless steels, (a) SUS430, (b) SUS304 and (c) SUS316L with pure Al.

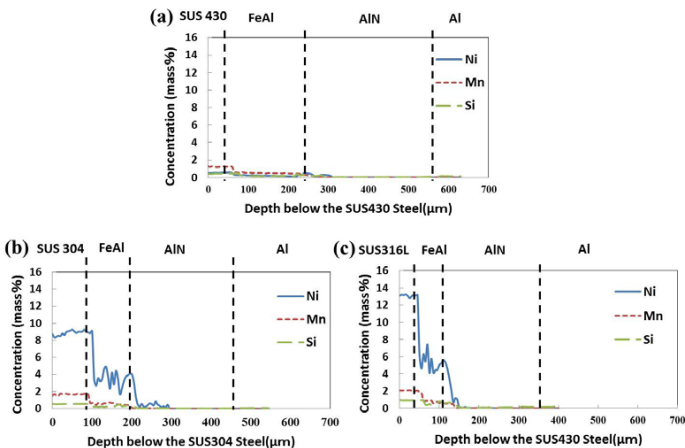


Fig. 6 EPMA analysis on the multilayer between the pure Al and various stainless steel, (a) SUS430, (b) SUS304 and (c) SUS316L.

As shown in Fig. 6, EPMA analysis was performed to investigate the amount of Ni, Si and Mn with behavior of the elements. Each graph shows the stainless steel, Fe-Al layer, AlN layer and aluminum area with the variation of each element. As a result from the EPMA, the Ni concentration was increased on the Fe-Al intermetallic compound layer with increasing the Ni concentration of the stainless steel such as the SUS304 and SUS316L. It means that the Ni was diffused from stainless steel to Fe-Al intermetallic compound. And it caused the thickness variation of Fe-Al compound layer. That is, the Ni concentration which was diffused from stainless steel hinders the diffusion of Al from the aluminum substrate to the stainless steel. Thus, it is believe that the thickness of Fe-Al compound layer is increased with decreasing the Ni concentration.

Conclusions

- 1) After barrel nitriding, the aluminum nitride layer (AlN) and Fe-Al intermetallic compound layers formed at the interface between aluminum and various stainless steels at 640 °C for 10hrs.
- 2) The total thickness of the multilayer decreased with increasing the Ni concentration of stainless steel. The thickness of the multilayer showed about 500 μm, 360 μm and 270 μm at the SUS430, SUS304 and SUS316L, respectively.
- 3) The hardness of the Fe-Al intermetallic compound and the aluminum nitride layer showed about 900 HV and 270HV, respectively. However, the significant changes in hardness were not found as different Ni concentration, except for the thickness.

Acknowledgments

This work was supported by Toyota Technological Institute.

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