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**Processing of *In-situ* Al-AlN Metal Matrix
Composites via Direct Nitridation Method**

Kwang Seon Shin, Yong-Seog Kim and Nack J. Kim

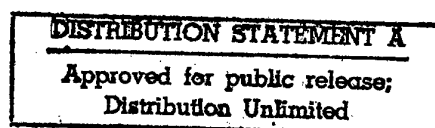
**Center for Advanced Aerospace Materials
Pohang University of Science and Technology
Pohang, Korea**

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**CENTER FOR ADVANCED AEROSPACE MATERIALS
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13. ABSTRACT (Maximum 200 words) The primary objective of the present research was to provide a fundamental understanding of the processing science necessary to fabricate the Aluminum Nitride (AIN) reinforced Aluminum (Al) composites via direct nitration of the Al alloy melt. From the thermodynamic considerations, it was found that reduction of oxygen content below the critical level in the reaction gas was crucial for the cussessful formation of AIN. It was possible to produce a considerable amount of AIN particles in the Al matrix at a reaction temperature as low as 900 "C" utilizing the in-situ nitration reaction process developed in the present study. The volume fraction of AIN increase almost linearly with increasing the magnesium (MG) content in the alloy and the reaction time. The shapes of AIN particles were found to be polygon, plate of rod-type, whose sizes were in the range from submicron to a few microns. From the present study, it is concluded that the new innovative in-situ nitridation process developed in the present study can be successfully applied for processing of high strength Al-AIN composites. DTIC QUALITY INSPECTED 4				
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PREFACE

This report presents the results of research performed from 1 September 1996 to 31 August 1997 by the Center for Advanced Aerospace Materials, Pohang University of Science and Technology (POSTECH) under the US AFOSR Grant F49620-96-1-0446 entitled "Processing of *In-situ* Al-AlN Metal Matrix Composites via Direct Nitridation Method." The intent of the study was to provide a fundamental understanding of the processing science necessary to fabricate the AlN-reinforced Al composites via direct nitridation of the Al alloy melt, and to evaluate innovative processing technique which have the potential to enhance productivity.

Prof. Nack J. Kim of the POSTECH was the Principal Investigator, with Prof. Kwang S. Shin of the Seoul National University and Prof. Yong S. Kim of the Hongik University as the Investigator. Dr. S. Fujishiro of the AOARD was the Program Manager for the grant.

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1. Introduction

High strength materials are of great interest in many aerospace and automotive applications from the viewpoint of their ability to save weight and to overcome other mechanical design limitation [1]. Metal matrix composites (MMCs) have a great potential for property improvements and have received a lot of research interest in recent years [2-4]. Particulate, whisker and continuous fiber-reinforced metal matrix composite (MMC) materials have usually been produced by powder metallurgy (P/M) process and squeeze casting process [1,5,6]. A number of problems associated with these conventional processing techniques in fabrication of MMCs tend to limit the widespread use of these materials in many applications [1]. These problems include high production costs, poor mechanical properties caused by weak interfacial strength between reinforcements and matrix materials, inadequate performance reliability and limitations on the sizes and shapes of the parts that can be produced. It is, therefore, necessary to develop new processing techniques for MMCs in order to overcome these problems.

In recent years, various new processing techniques have been developed for the production of composite materials utilizing the *in-situ* synthesis methods [7-14]. Several *in-situ* processing techniques including directional solidification, XD- and Lanxide processes have a potential for fabrication of MMCs at a lower cost and better performance [15,16]. Since the reinforcements and matrix materials are produced simultaneously during the *in-situ* processing of composite materials, mechanical properties can be significantly improved as a result of better interfacial strength [13].

The Lanxide process, which has been developed mainly for the processing of ceramic matrix composites (CMCs), involves the oxidation of a molten metal under gaseous oxidant (for example, the oxidation reaction of a molten aluminum alloy in oxidizing gas to form alpha-aluminum oxide matrix reinforced by metallic aluminum) [17]. Under the proper processing conditions, the reaction could proceed to produce CMCs with three-dimensionally interconnected aluminum channel. This process, however, requires higher reaction temperatures ($>1100^{\circ}\text{C}$) and longer reaction time (12~24 hours) than conventional processing techniques.

In recent years, there have been attempts to produce aluminum nitride (AlN) or AlN composites utilizing the reaction between the molten Al and the reaction gas [16]. It has been found that thin films of nitride and/or oxide are formed at the surface of the molten metal in the initial stage of the reaction. These films could retard the further progress of the reaction front. Therefore, it requires high reaction temperature and long reaction time for the successful formation of a significant amount of AlN. Furthermore, the effects of various processing variables such as alloying element, reaction gas,

processing temperature and time on the formation of AlN have not yet been clearly understood.

The primary objective of the present research is to provide a fundamental understanding of the processing science necessary to fabricate the AlN-reinforced Al composites via direct nitridation of the Al alloy melt. The effects of processing variables including reaction temperature and time, reaction gas and alloying element on the fraction, morphology and distribution of AlN were investigated. Furthermore, it was attempted to develop an innovative processing technique for the fabrication of AlN-reinforced Al composites via direct nitridation of the molten Al at lower temperature than that required for the conventional processing techniques. In the present study, the reaction gas was continuously blown into the stirred Al melt in order to maximize the nitridation reaction.

2. Thermodynamic Considerations

The molten aluminum can react with nitrogen bearing gas and aluminum nitride (AlN) can be formed according to the following reaction expressed by Eqn. (1). Figure 1 shows the temperature dependence of the Gibbs' free energy change for nitridation reaction. Also shown in Fig. 1 are the Gibbs' free energy changes for some oxidation reactions that can occur depending upon the reaction gas and alloy composition.



Thermodynamically, the nitridation of aluminum is an exothermic process and is energetically favorable over a wide temperature range. However, it has been reported that the nitridation of pure aluminum generally requires the reaction temperature higher than 1200°C. This result indicates that there may be other important factors that affect the nitridation of the aluminum melt. First possible explanation of the experimental result is that the nitridation of the aluminum melt may largely be controlled by the kinetic factors. Although the nitridation reaction is thermodynamically favorable, the reaction may proceed very slowly so that it requires long time for the formation of AlN at low temperatures. Second possible explanation is the formation of other compounds such as oxides that may retard the nitridation reaction by acting as

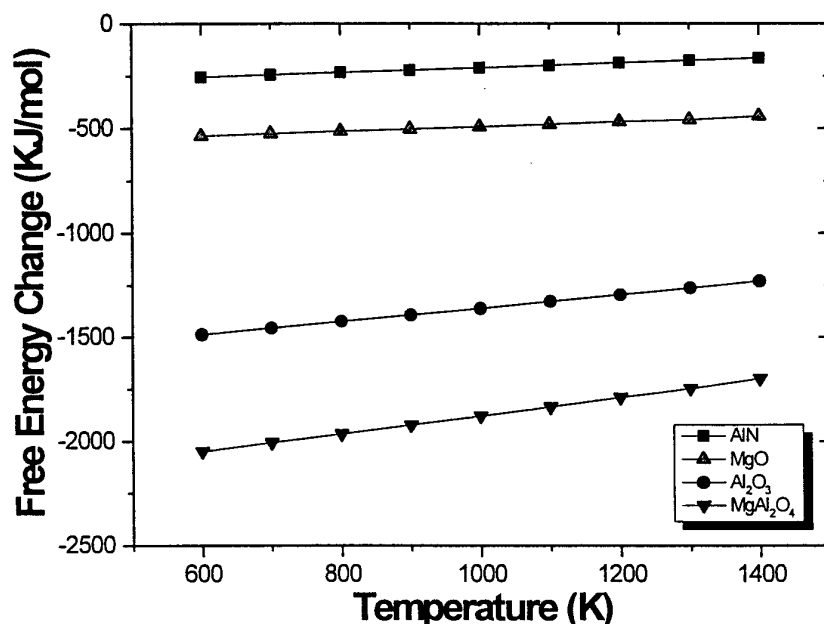
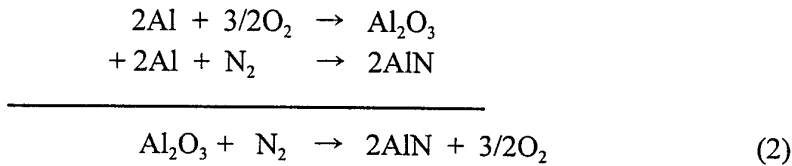


Figure 1. Gibbs' free energy change with temperature for the formation of AlN and oxides.

reaction barriers. The Gibbs' free energy changes for some oxidation reactions shown in Fig. 1 indicate that these oxidation reactions are thermodynamically more favorable than the nitridation of aluminum.

The activation energies of nitridation reactions are reported to be 93.4 KJ/mol in the A380.1 alloy [7] and 20.8 KJ/mol in the Al-4wt.%Mg alloy [8]. These results indicate that the alloying element can significantly affect the nitridation reaction of aluminum alloys. However, the exact role of alloying element in the nitridation reaction has not yet been clearly understood. In the present study, the effect of alloying element will be examined in the *in-situ* nitridation process.

As described previously, the formation of Al_2O_3 and MgO is thermodynamically more favorable than that of AlN. Therefore, the effect of oxygen on the nitridation reaction should be considered for effective formation of AlN in the *in-situ* process. The reactions for the formation of Al_2O_3 and AlN can be expressed by the following Eqn. (2).



For the above reaction, the critical partial pressure of oxygen in the nitrogen gas can be calculated using Eqn. (3) for the formation of Al_2O_3 instead of AlN in the molten Al.

$$P_{O_2} = \left[\frac{a_{Al_2O_3} P_{N_2}}{a_{AlN}} \exp\left(-\frac{\Delta G_f^\circ}{RT}\right) \right]^{\frac{2}{3}} \quad (3)$$

Assuming that the activities of solid AlN and Al_2O_3 are unity, the critical partial pressure of oxygen in 1 atm nitrogen gas was calculated to be 2.22×10^{-27} atm for the formation of Al_2O_3 instead of AlN at a reaction temperature of 1200K. It is, therefore, critical to remove oxygen completely from the reaction gas in order to form AlN without forming Al_2O_3 .

3. Experimental Procedures

3-1. Thermogravimetric Analyses

Thermogravimetric analyses (TGA) were carried out in order to investigate the effect of Mg on the nitridation reaction and determine the optimum temperature for the *in-situ* nitridation process. High purity Al and Mg powders with sizes of -325 mesh were used as starting materials for the TGA specimens. A cylindrical pellet was prepared from one gram of mixed powders with a desired composition. The contents of Mg in the TGA specimens were 0, 1, 4, 6 and 10 wt.%. The specimens were heated at a rate of 10 °C/min from 100 °C to 1000 °C while flowing the N₂ + 5%H₂ (>99.999%) gas at a rate of 500 cc/min.

Isothermal thermogravimetric analyses were also conducted in order to examine the effect of Mg addition on the nitridation reaction at 900 °C. The specimens were heated to 100 °C from room temperature at a rate of 5 °C/min and then to 900 °C at a rate of 10 °C/min. The specimens were held for 5 hours at 900 °C.

The weight of the TGA specimen was continuously measured with an electronic balance connected to a computer in order to determine the weight change during the nitridation reaction. Figure 2 shows a schematic diagram of the TGA system.

After the TGA experiments, the specimens were analyzed by X-ray diffraction (XRD) in order to identify the reaction products. The volume fraction of AlN in the reaction product was determined in a quantitative manner.

3-2. *In-situ* Nitridation Processes

Table 1 shows the chemical compositions of pure Al (ALCAN Co.: 99.7%) and Mg (Norsk Hydro Co.: 99.8%) ingots used as starting materials in the present study. The Al-25wt.%Si mother alloy was also used in order to prepare the aluminum melts with desired chemical compositions.

Table 1. Chemical compositions of the starting materials.

Alloy	Mg	Fe	Cr	Si	Ni	Al
Al	0.001	0.11	0.001	0.04	0.005	bal.

Alloy	Al	Fe	Cr	Si	Ni	Mg
Mg	0.01	0.12	0.001	0.03	0.006	bal.

The ingots were initially cut to chunks with appropriate sizes. The chunks were ultrasonically cleaned in acetone for removal of surface contaminants and then being placed in a crucible in the reaction system. The charge weight was 1.5 kg per batch.

The alloy chunks were heated at a rate of $7^{\circ}\text{C}/\text{min}$ in an argon atmosphere. Once the temperature of the molten alloy reached at 750°C , mechanical stirring was carried out for 10 minutes with a stirrer in order to obtain uniform temperature and composition of the alloy melt. After homogenization the alloy melt was heated to a desired reaction temperature and the reaction gas was blown into the middle of the alloy melt. In the present study, the $\text{N}_2 + 5\%\text{H}_2$ ($>99.999\%$) gas was used as the reaction gas in order to

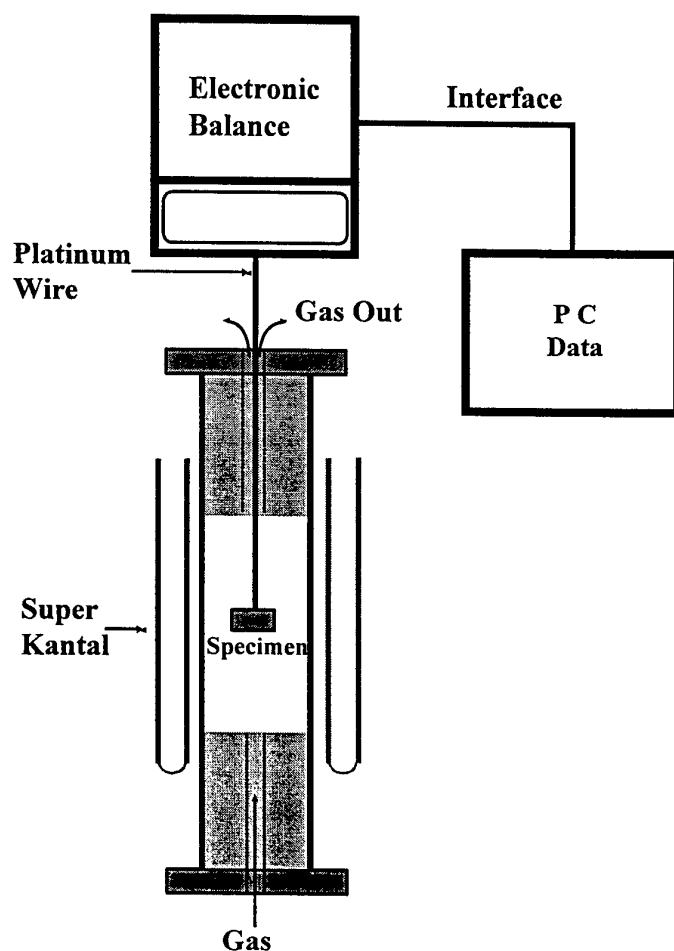


Figure 2. Schematic diagram of the thermogravimetric analysis system.

minimize the oxidation reaction. The reaction gas was purified and dried before it was supplied to the alloy melt. After the preliminary experiments with various flow rates of the reaction gas, a flow rate of 400 cc/min. was chosen as the optimum rate in the present study. The alloy melt was continuously stirred at a rate of 300 rpm during the *in-situ* nitridation reaction in order to promote the reaction by producing small gas bubbles and uniformly distribute the formed AlN particles. The reaction times were varied from 0 to 32 hours. Figure 3 shows a schematic diagram of the reaction system used in the present study. A schematic diagram of the *in-situ* nitridation reaction is shown in Fig. 4. The reaction gas was supplied to the middle of the alloy melt through a hole that was placed at the center of the stirrer.

After the desired reaction time, the reacted melt was cooled to 750°C and then poured into a graphite mold. In order to achieve uniform distribution of AlN particles in the matrix, the graphite mold was preheated at 180°C.

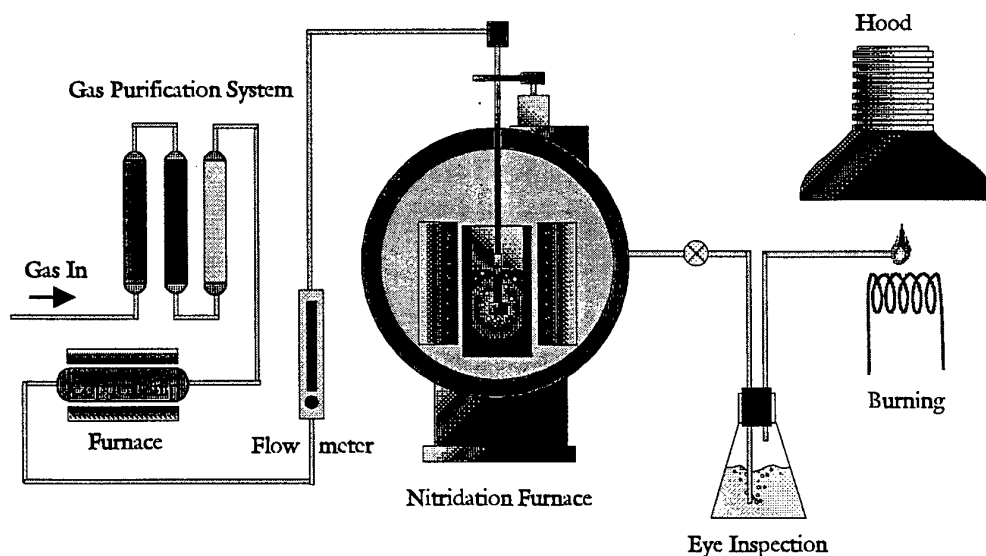


Figure 3. Schematic diagram of the experimental system used in the present study.

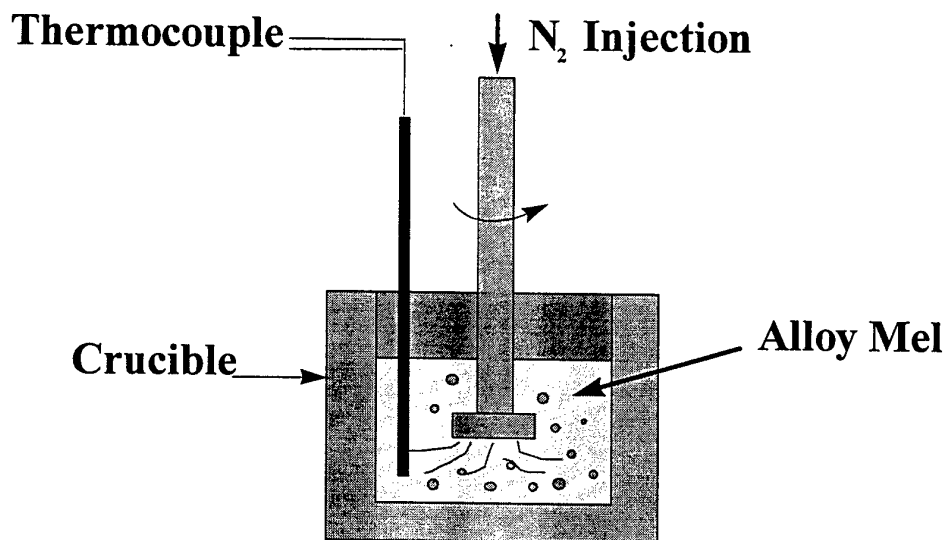


Figure 4. Schematic diagram of the *in-situ* nitridation reaction.

3-3. Microstructure Analyses

The microstructures of the reacted specimens were examined by optical and scanning electron microscopes (JEOL, JSM-840A). The fraction and distribution of the reaction products were examined by an image analysis system (BMI Plus). The specimens for microstructure observation were cut from the predetermined locations of the ingots and examined after being etched in a 0.5% HF solution for 30~60 seconds. The sizes and morphologies of the reaction products were examined by a scanning electron microscope. In order to examine the size and the shape of the reaction products in a detailed manner, the reaction products were extracted from the ingots after nitridation reaction by leaching out the Al matrix and examined by scanning electron microscopy at high magnifications.

3-4. X-ray Diffraction Analyses

In order to determine the reaction products after the nitridation experiments, X-ray diffraction (XRD) analyses were carried out on the specimens taken from the predetermined positions of the ingots after nitridation reaction with a Mac Science Model M18XHF-SRA machine. The XRD results were obtained with a Cu target at an

accelerating voltage of 50 kV, a current of 100 mA and a scanning speed of 10 degree/min. In order to minimize the effect of grain orientation on X-ray peak intensities, fine powder samples were used for the XRD analyses that were produced from the ingots obtained from the nitridation reaction. The volume percent of AlN in the ingot was determined in a quantitative manner by an internal standard method. The reference curve was produced from the XRD data obtained from the standard samples with known compositions of AlN.

4. Results and Discussion

4-1. Thermogravimetric Analyses

Table 2 shows the weight changes of the specimens with various Mg contents after the isothermal thermogravimetric analyses (TGA) at 900°C for 5 hours. It was found that the weight of the specimen increased after the nitridation reaction regardless of Mg content. However, the weight gain appeared to increase with increasing Mg content in the specimen. In order to compare the weight gain in different specimens, the percent weight gain was defined according to Eqn. (4) and the results are shown in Table 2.

Weight Gain (%)

$$= (\text{Final Weight} - \text{Initial Weight}) / (\text{Initial Weight}) \times 100 \quad (4)$$

From the experimental results, the percent weight gain of the specimen was found to increase with increasing Mg content. The results of XRD analyses on the TGA specimens showed that the reaction products contained AlN as well as oxides as shown in Table 2 except the pure Al specimen that did not show any significant AlN peak. It was considered that the weight gain of the specimen was caused by the formation of AlN and some oxides that increased with increasing Mg content. It is, therefore, considered that Mg increases the rate of the nitridation reaction and promotes the formation of AlN.

Table 2. Results of the isothermal TGA tests for 5 hours at 900°C.

Alloy System	Initial Weight (g)	Final Weight (g)	Percent Weight Gain (%)	Results of XRD Analyses
Al-0wt.% Mg	0.98057	0.99370	1.34	Al ₂ O ₃
Al-1wt.% Mg	0.99349	1.11043	11.77	AlN, MgO, Al ₂ O ₃
Al-4wt.% Mg	1.00690	1.15068	14.28	AlN, MgO
Al-6wt.% Mg	0.95133	1.13149	18.94	AlN, MgO
Al-10wt.% Mg	0.00219	1.22355	22.09	AlN, MgO

Figure 5 shows the changes in the percent weight gain of the specimen during the isothermal TGA tests at 900°C for 5 hours. Without Mg addition, the weight of the specimen showed very small increase with exposure time to the reaction gas. However,

with Mg addition, the weight increased significantly as soon as the reaction temperature increased. The weight gain after 5 hours of exposure to the reaction gas at 900°C increased with increasing Mg content in the specimen. These results clearly indicated that Mg had a significant effect on the nitridation of the molten Al.

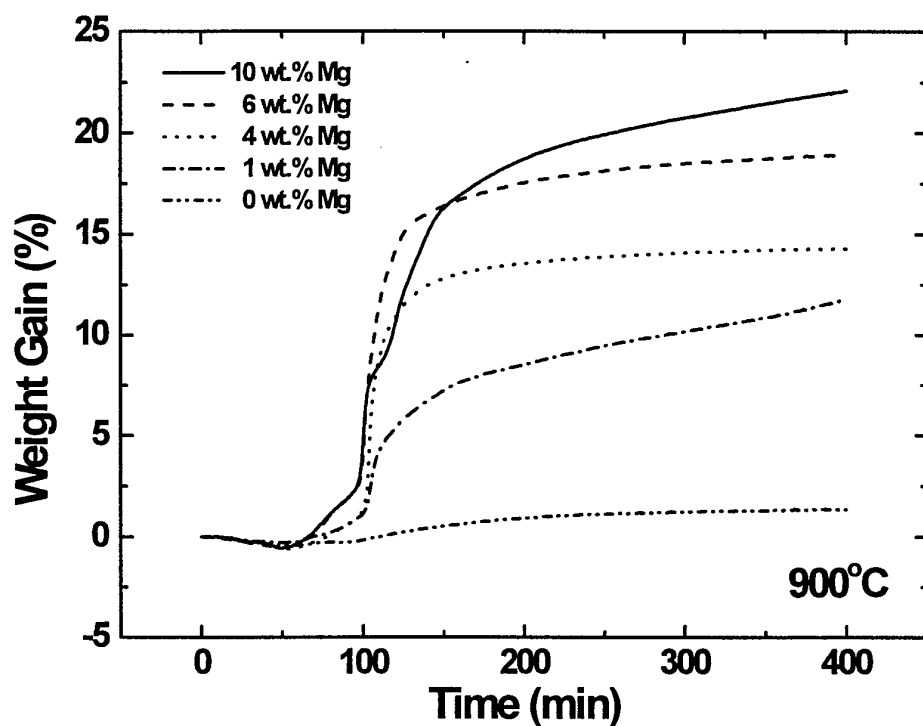


Figure 5. Weight gain of Al-Mg alloys during the isothermal TGA tests at 900°C.

Figures 6 and 7 show the results of the TGA on Al-4wt.%Mg and Al-10wt.%Mg alloys in the temperature range from 100°C to 1100°C. The derivatives of the weight gains are also shown in Figs. 6 and 7. The result obtained from the Al-4wt.%Mg alloy showed the rapid increase in weight gain at about 860°C and the maximum increase in weight gain was obtained at 906°C. On the other hand, the Al-10wt.%Mg alloy showed the rapid increase in weight gain at about 830°C, which was about 30°C lower than that

found in the Al-4wt.%Mg alloy. The maximum increase in weight gain in the Al-10wt.%Mg alloy was obtained at 873 °C, which was also about 30 °C lower than that found in the Al-4wt.%Mg alloy. These results also indicated that Mg increased the reaction rate between the Al melt and the reaction gas and promoted the formation of AlN in the present study. Based upon these experimental results, the reaction temperature for the *in-situ* nitridation experiments was chosen as 900 °C.

4-2. *In-situ* Nitridation of Al-Mg Alloys

In the present study, it was attempted to form aluminum nitride (AlN) by *in-situ* nitridation reaction between the molten Al alloy and the reaction gas. After the alloy melt was heated to the desired reaction temperature, the reaction gas was blown into the melt. The reaction temperature was chosen to be 900 °C at which the weight gain

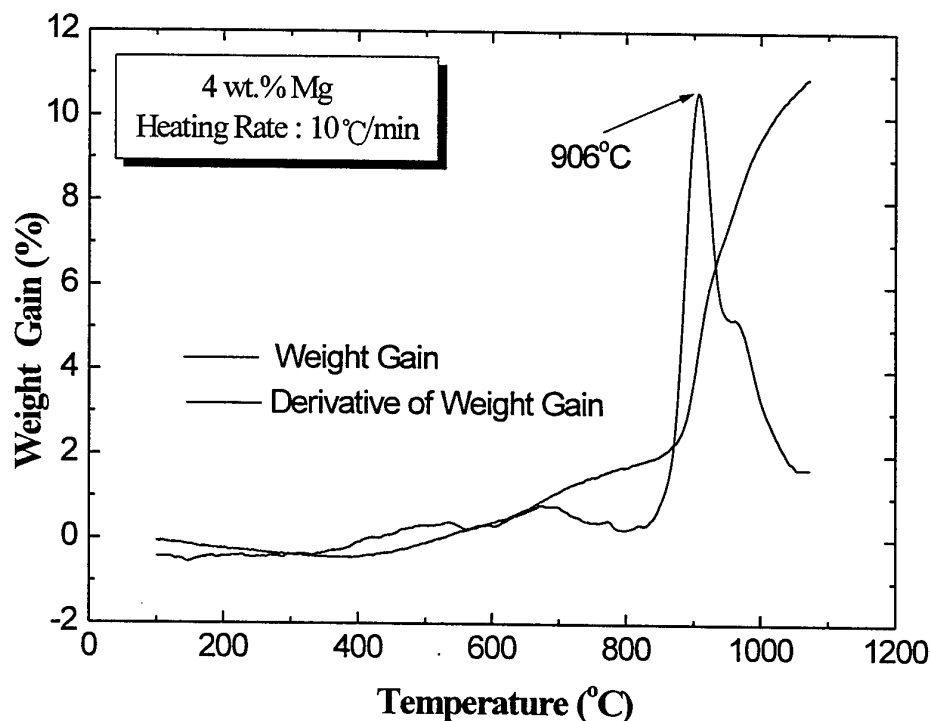


Figure 6. Weight gain of the Al-4wt.%Mg specimen during the TGA tests.

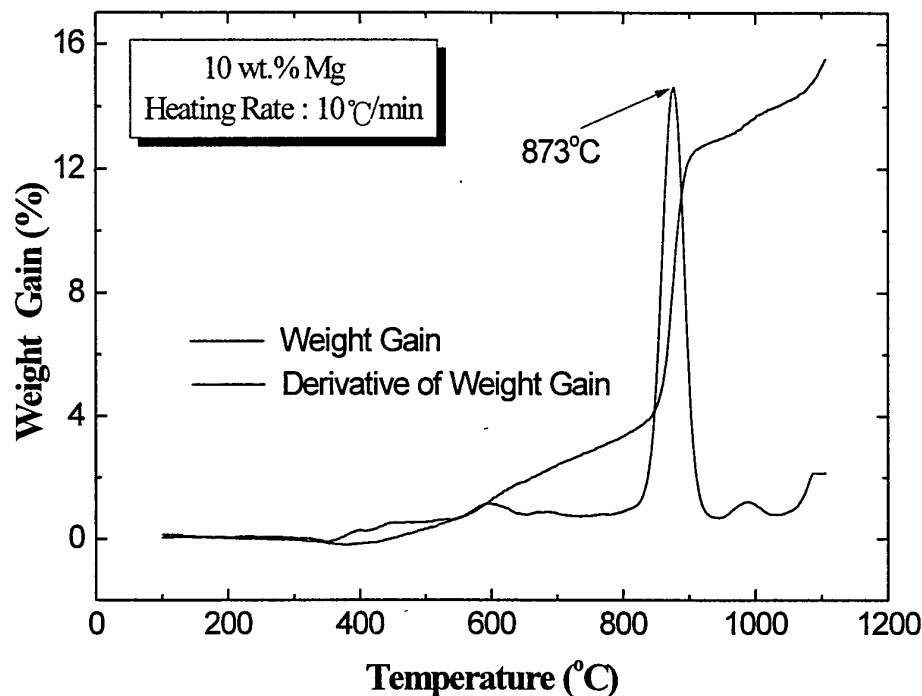


Figure 7. Weight gain of the Al-10wt.%Mg specimen during the TGA tests.

increased dramatically during the thermogravimetric analyses (TGA). The $N_2 + 5\%H_2$ (>99.999%) gas was used as the reaction gas in order to minimize the oxidation reaction. The reaction gas was purified and dried before it was supplied to the alloy melt. A flow rate of 400 cc/min was used in the present study. The alloy melt was continuously stirred at a rate of 300 rpm during the reaction in order to promote the formation of AlN particles by producing small gas bubbles and uniformly distribute the formed AlN. The reaction times were varied from 0 to 32 hours.

As described previously, the formation of Al_2O_3 and MgO is thermodynamically more favorable than that of AlN. The critical partial pressure of oxygen in 1 atm nitrogen gas was calculated to be 2.22×10^{-27} atm for the formation of Al_2O_3 instead of AlN at a reaction temperature of 1200K. It is, therefore, critical to remove oxygen and moisture as much as possible from the reaction gas in order to promote the formation of AlN instead of oxides during the *in-situ* process. A gas purification system shown in

Fig. 8 was used in order to minimize the amounts of oxygen and moisture in the reaction gas. The reaction gas was passed through dryerite, activated carbon and alumina and then copper chips heated to 500 °C. Furthermore, 5 vol.% hydrogen was added to the reaction gas in order to minimize the oxidation reaction. As a result, the oxidation reaction could be significantly suppressed. Oxide particles were hardly found in the ingots obtained after the nitridation reaction. The XRD results obtained from the ingots also did not show any significant peaks corresponding to oxides. Another evidence of effective removal of oxygen from the reaction gas was obtained from the analysis of the particles deposited on the wall of the reaction furnace. Figure 9 shows the results of the XRD analysis of the powders collected from the furnace wall. The results showed mainly the peaks corresponding to Mg and the X-ray peaks for MgO decreased significantly.

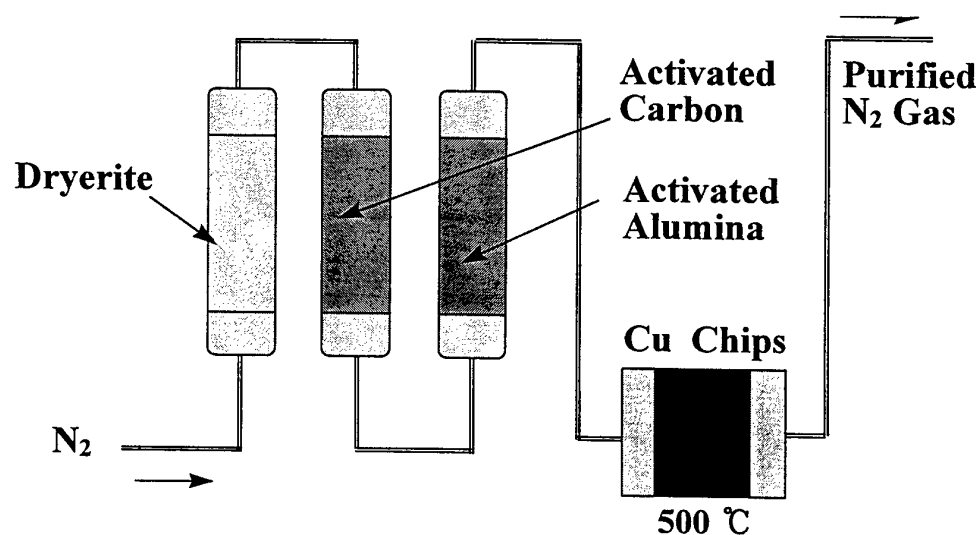


Figure 8. Schematic diagram of the gas purification system.

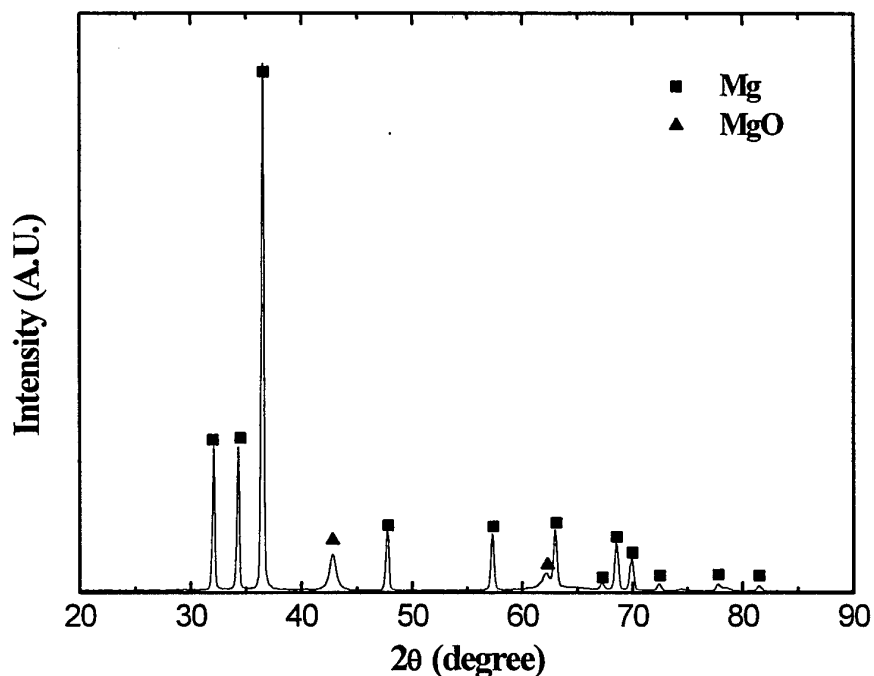


Figure 9. XRD pattern of the vaporized powders on furnace wall.

During the *in-situ* nitridation reaction of the Al-Mg alloy melt, it was found that Mg had a tendency to evaporate because of its high vapor pressure. The furnace wall. Therefore, the Mg contents of the Al-Mg alloys decreased with evaporation of Mg was confirmed by the deposition of Mg powders on the increasing reaction time. Table 3 shows the Mg contents of the specimens after the nitridation reaction analyzed by ICP.

As can be seen in the Table 3, the Mg content in the specimen decreased from the initial value after the nitridation reaction. The amount of decrease in Mg content also increased with reaction time. These results indicated that Mg in the alloy melt evaporated during the nitridation reaction due to its high vapor pressure at the reaction temperature.

The microstructure of the ingot obtained from pure Al melt did not show any significant change after nitridation reaction at 900°C regardless of reaction time.

However, the ingots obtained from the Al-Mg alloy melts clearly showed the particles that were considered to be the reaction products.

Table 3. The Mg content of the specimen after the nitridation reaction.

Initial Composition	Nitridation Reaction Time (hrs)	Final Mg Content (wt.%)
Al-4wt.%Mg	4	3.48
Al-4wt.%Mg	8	2.97
Al-4wt.%Mg	16	2.0
Al-4wt.%Mg	32	1.94
Al-10wt.%Mg	4	8.41
Al-10wt.%Mg	8	8.17
Al-10wt.%Mg	16	6.32
Al-10wt.%Mg	32	4.58
Al-20wt.%Mg	4	18.95
Al-20wt.%Mg	8	17.3
Al-20wt.%Mg	16	16.3
Al-20wt.%Mg	32	9.97

Figures 10, 11 and 12 show the optical micrographs of the ingots after various reaction times that were obtained from the alloys initially containing 4, 10 and 20 wt.% Mg, respectively. All micrographs showed small black particles that were uniformly distributed in a macroscopic scale. These black particles were considered to be the reaction products of the nitridation reaction and confirmed by the X-ray diffraction (XRD) analysis as AlN particles. In a microscopic scale, however, most AlN particles were found to segregate at grain boundaries. It was believed that the reaction formed AlN particles were pushed to and segregated at grain boundaries during solidification because a material with low melting point at grain boundaries solidified in the last stage of solidification.

The optical micrographs shown in Fig. 10 were obtained from the ingots after nitridation reaction of the Al-4wt.%Mg alloy. After 4 hours of reaction, small black

particles began to appear in the optical micrograph as shown in Fig. 10 (a). The number of particles increased as the reaction time increased from 4 to 32 hours. The particles were also found to distribute rather uniformly throughout the ingot in a macroscopic scale. Figures 11 (a)~(d) show the optical micrographs obtained from the ingots after nitridation reaction of the Al-10wt.%Mg alloy for various reaction times. The micrograph obtained from the ingot reacted for 4 hours showed only small amount of AlN particles as shown in Fig. 11 (a). The amount of AlN particles, however, increased significantly with reaction time. A quantitative analysis of the XRD pattern obtained from the ingot reacted for 32 hours indicated that the volume percent of AlN was almost 8%. The average grain size of the ingot also appeared to decrease with increasing reaction time, indicating that the AlN particles acted as barriers to grain boundary motion and suppressed grain growth.

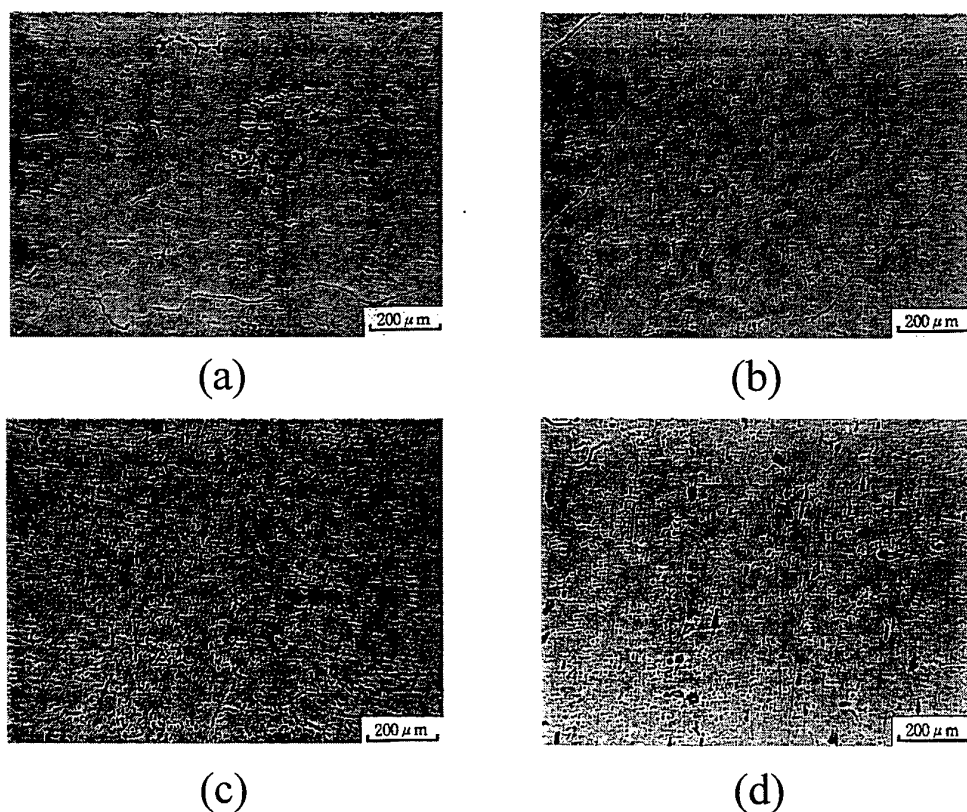


Figure 10. Optical micrographs of the nitrided specimens with reaction time (Al-4wt.%Mg alloys): (a) 4 hours (b) 8 hours (c) 16 hours and (d) 32 hours.

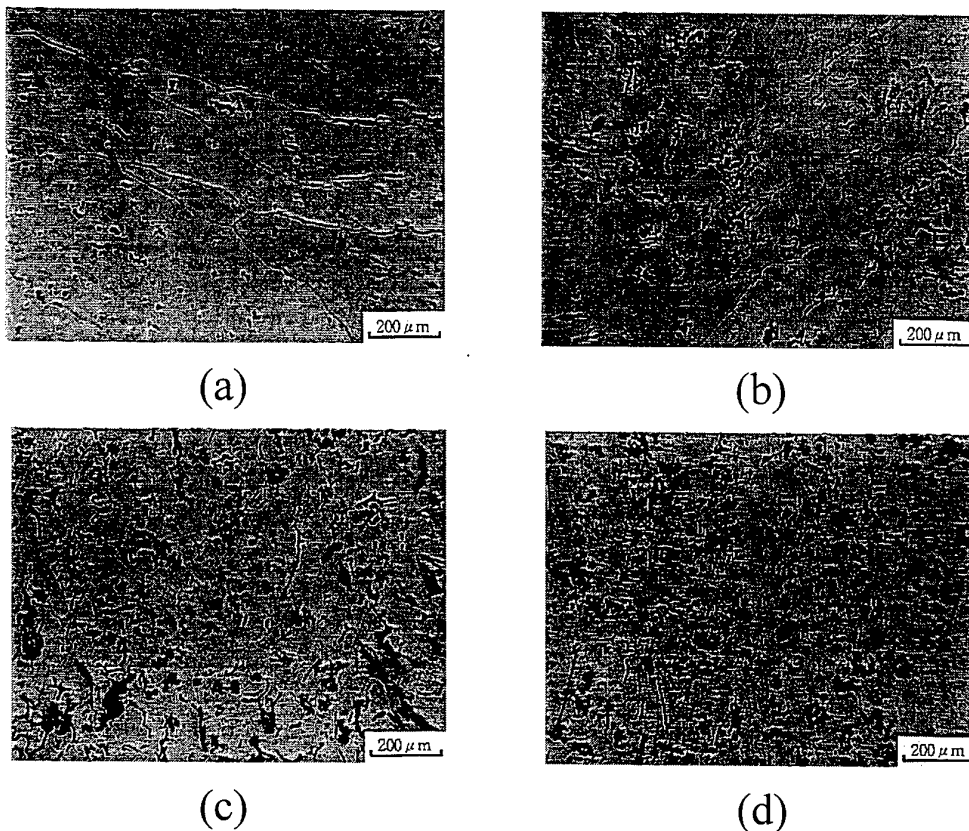


Figure 11. Optical micrographs of the nitrided specimens with reaction time (Al-10wt.%Mg alloys): (a) 4 hours (b) 8 hours (c) 16 hours and (d) 32 hours.

Figures 12 (a)~(d) show the optical micrographs obtained from the ingots after nitridation reaction of the Al-20wt.%Mg alloy. The micrograph obtained from the ingot reacted only for 4 hours clearly showed a considerable amount of AlN particles as shown in Fig. 12 (a). The micrograph also clearly showed the decrease in grain size and the presence of the second phase that was believed to be the Al_3Mg_2 compound. The amount of AlN particles also increased significantly with reaction time as shown in Fig. 12.

The examination of the microstructures of the ingots obtained from the Al-Mg alloys clearly showed the effect of Mg addition on the nitridation reaction. In the present

study, the *in-situ* nitridation reaction of the pure Al melt hardly produced any AlN particles at 900°C regardless of reaction time. The result was in agreement with the previously reported results. It has been reported that the nitridation of pure aluminum generally requires the reaction temperature higher than 1200°C [8,14]. The amount of reaction formed AlN, however, increased significantly with increasing Mg content in the alloy melt, indicating a significant effect of Mg on the formation of AlN even in the case of present *in-situ* nitridation process. The role of Mg in the present *in-situ* nitridation process could be different from that in the DIMOX process [13,17]. Further study is clearly required for the identification of the exact role of Mg.

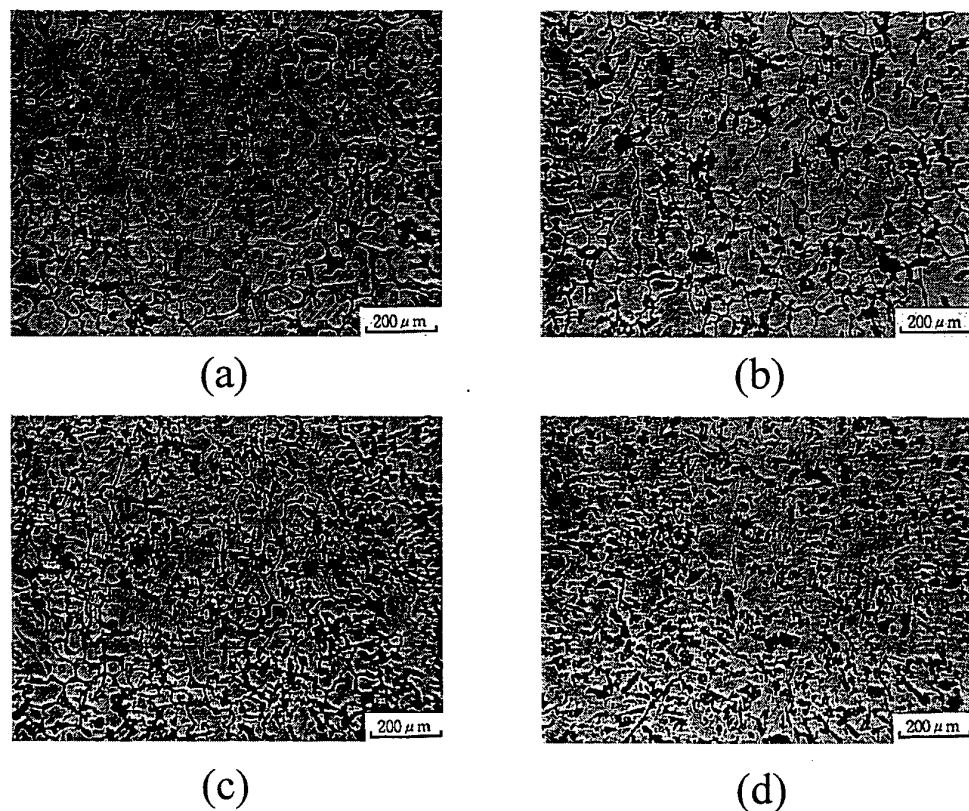


Figure 12. Optical Micrographs of the nitrided specimens with reaction time (Al-20wt.%Mg alloys): (a) 4 hours (b) 8 hours (c) 16 hours and (d) 32 hours.

In order to identify and determine the volume fraction of the reaction products, X-ray diffraction (XRD) analyses were carried out on the specimens taken from the predetermined positions of the ingots after nitridation reaction. The ingot generally had a cast microstructure with preferred grain orientation and this could significantly affect the X-ray peak intensities of specific crystallographic planes. It was, therefore, necessary to prepare the fine powder samples for the XRD analyses from the ingots for the quantitative analysis. All prepared powder samples were subsequently annealed for the removal of residual stress caused by cold work.

Figure 13 shows the XRD pattern of the specimen taken from the ingot obtained by nitridation reaction at 900 °C from the alloy with an initial composition of Al-20wt.%Mg. The ingot was prepared with a reaction time of 32 hours. The XRD pattern shows mainly X-ray peaks corresponding to Al and AlN as indicated in Fig. 13. The crystal structure of AlN was identified to be hexagonal from the XRD analysis.

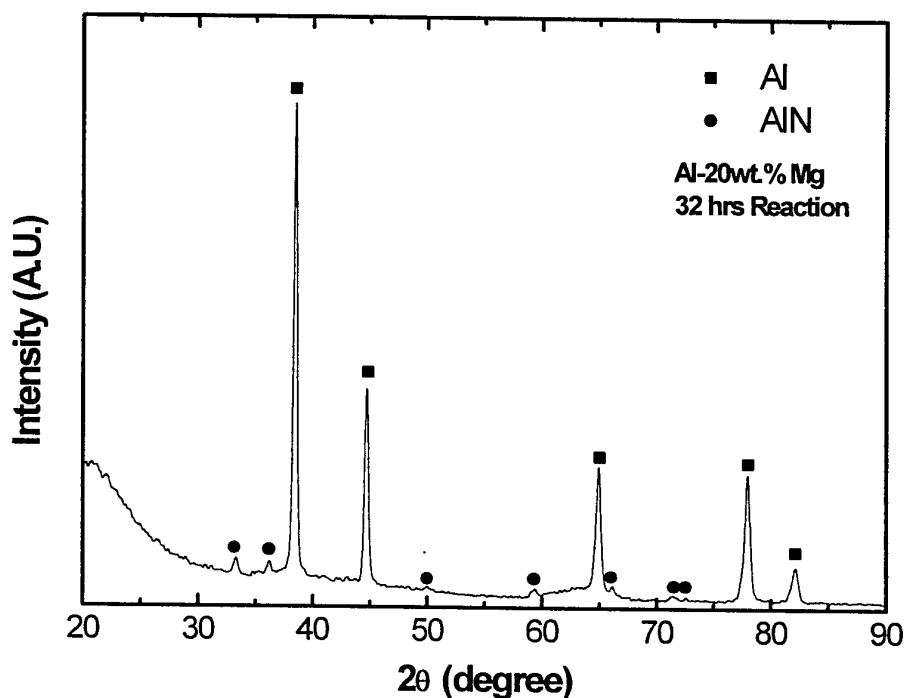


Figure 13. XRD pattern of the Al-20wt.%Mg specimen after 32hrs reaction.

The amount of AlN particles in the nitrided specimen was determined in a quantitative manner by an internal standard method using the ratio of the integral intensity of the AlN (0001) peak and that of the Al (200) peak. The reference curve for the quantitative analysis of the AlN content was obtained from the XRD analyses of the mixed powders of Al and AlN with known chemical compositions. The volume percent of AlN was calculated from the chemical composition of the mixed powder and plotted against the integral peak intensity ratio ($I_{\text{AlN}}/I_{\text{Al}}$). Figure 14 shows the reference curve for the quantitative XRD analysis of AlN content and the slope between the volume percent of AlN and the integral intensity ratio ($I_{\text{AlN}}/I_{\text{Al}}$) was determined to be 148.5. The AlN contents of the all nitrided ingots were determined by the comparison of the XRD results of the powder samples and the reference curve. The amounts of AlN in the ingots prepared from the alloy initially containing 4 wt.%Mg were very small and less than 1 vol.% after nitridation reaction at 900 °C regardless of reaction time in the present *in-situ* nitridation process. However, the effect of Mg addition on the formation of AlN was clearly observed in the ingots prepared from the alloys initially containing 10 and 20 wt.% Mg.

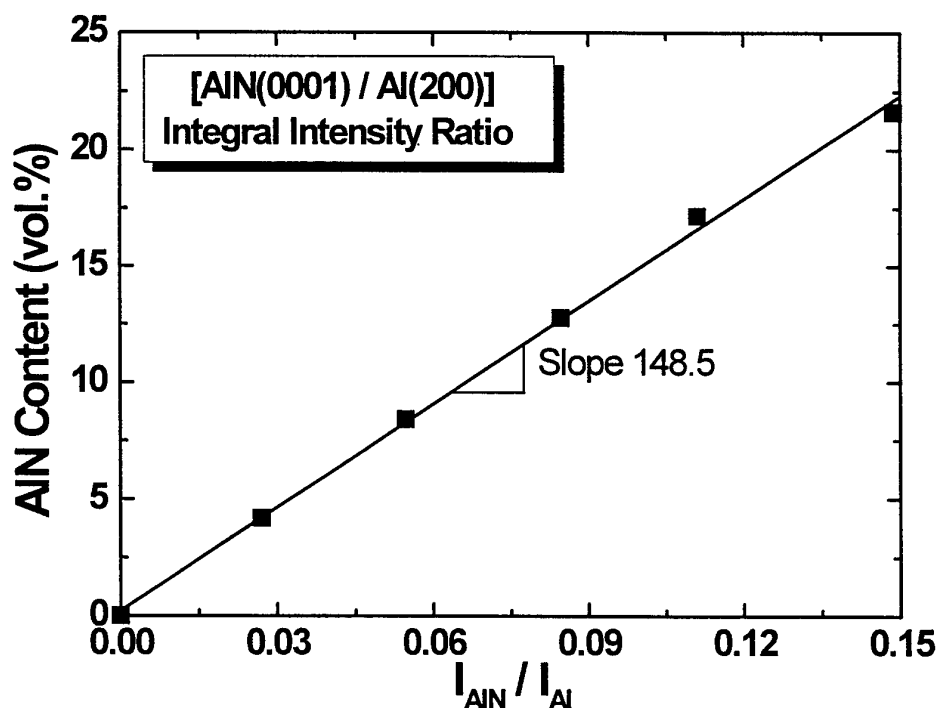


Figure 14. Reference curve for the quantitative XRD analysis.

The changes in the volume fraction of AlN particles are shown as a function of reaction time in Figs. 15 and 16 for the ingots obtained by nitridation reaction at 900 °C from the alloys with initial compositions of Al-10wt.% Mg and Al-20wt.%Mg, respectively. In both cases, the volume fraction of AlN increased almost linearly with increasing the reaction time. In case of the ingots prepared from the alloy initially containing 10 wt.%Mg, the AlN content was almost 8 vol.% after 32 hours of reaction. In case of the alloy initially containing 20 wt.%Mg, more than 10 vol.% of AlN was obtained after the nitridation reaction at 900 °C for 32 hours. From these experimental results, it could be concluded that the amount of AlN increased with initial Mg content in the alloy and nitridation time. It was considered that Mg promoted the formation of AlN in the *in-situ* nitridation process employed in the present study either by acting as catalyst or by reducing the reaction barriers for the nitridation reaction. These results were consistent with the results obtained from the thermogravimetric analyses.

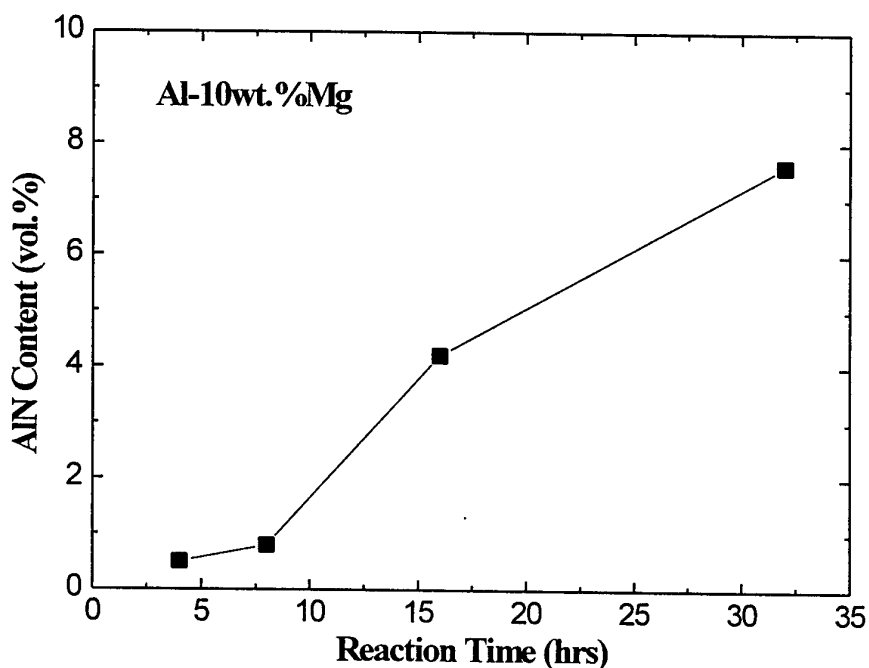


Figure 15. AlN content in the Al-10wt.%Mg specimen with reaction time.

The morphologies of AlN particles in the ingots obtained from nitridation reaction were examined by scanning electron microscopy (SEM). Figures 17, 18 and 19 show the scanning electron micrographs of AlN particles formed in the ingots after various reaction times that were prepared from the alloys initially containing 4, 10 and 20 wt.% Mg, respectively. The shapes of AlN particles were found to be polygon, plate or rod-type. The sizes of AlN particles were found to be in the range from submicron to a few microns. Both the size and the shape of AlN particles did not appear to change significantly by the initial composition of the alloy or reaction time in the present study.

In order to examine the size and the shape of AlN particles in a detailed manner, the reaction products were extracted from the ingots by leaching out the Al matrix and examined by scanning electron microscopy at high magnifications. Figures 20 and 21 show the scanning electron micrographs of the extracted reaction products from the ingots that were prepared from the alloys initially containing 10 and 20 wt.% Mg, respectively. The shapes of AlN particles were again confirmed to be polygon, plate or rod-type.

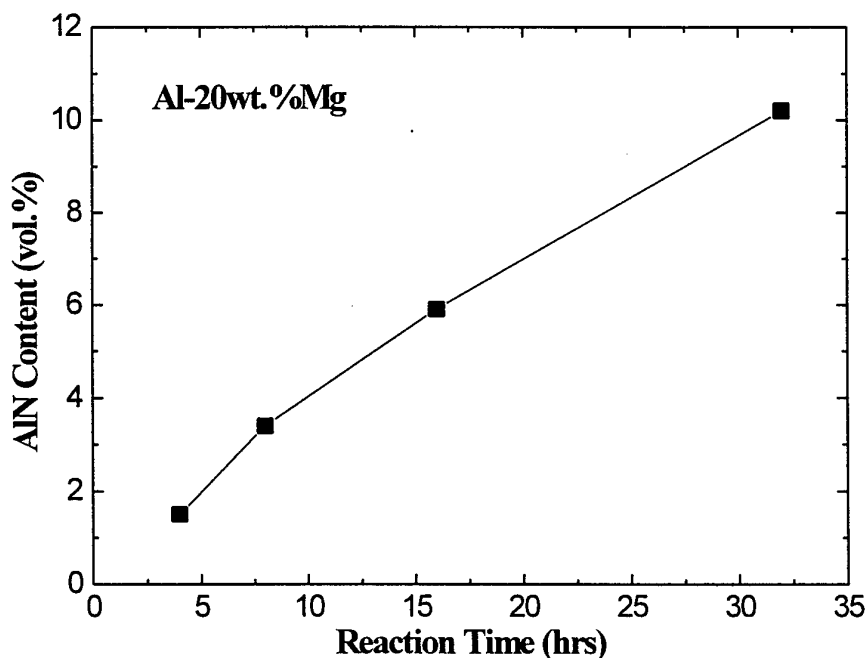
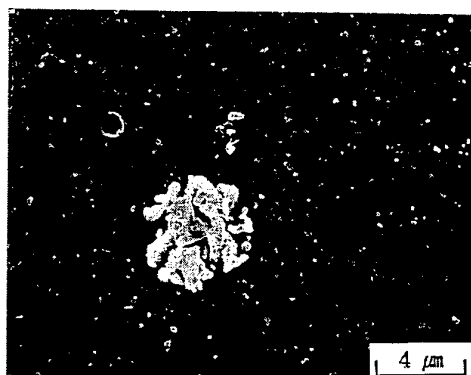
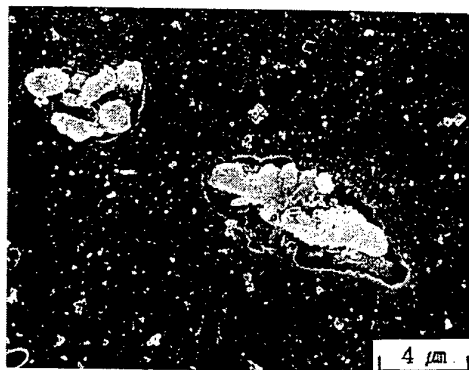


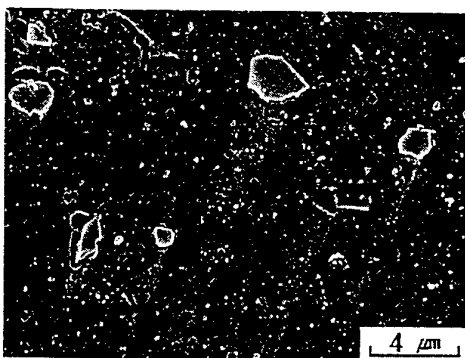
Fig. 16. AlN content in the Al-20wt.%Mg specimen with reaction time.



(a)



(b)

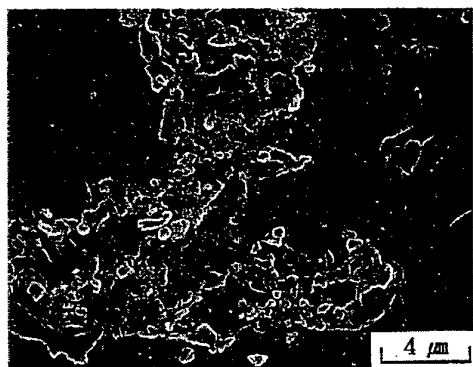


(c)

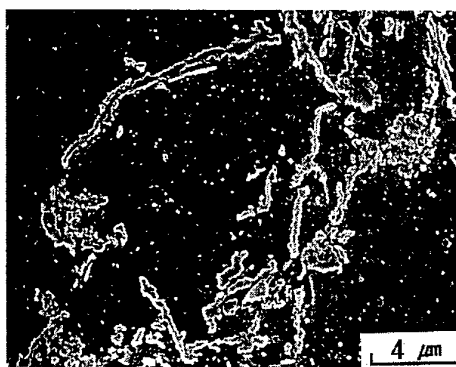


(d)

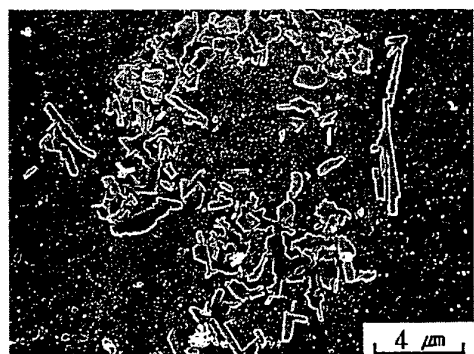
Figure 17. Particle morphologies in the Al-4wt.%Mg specimens after various reaction times: (a) 4 hours (b) 8 hours (c) 16 hours and (d) 32 hours.



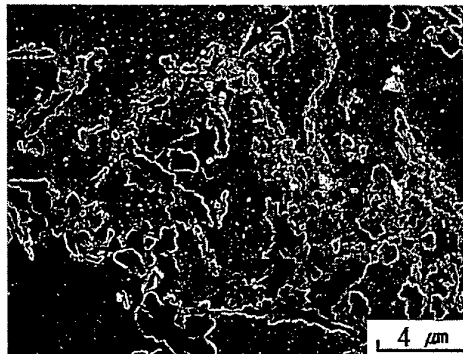
(a)



(b)

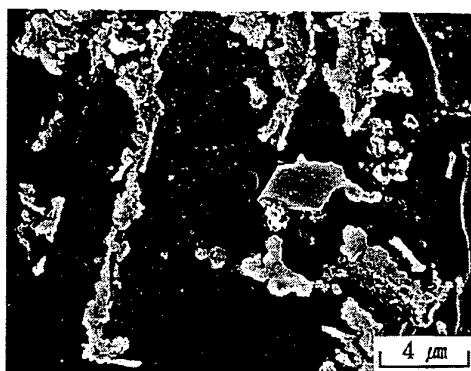


(c)



(d)

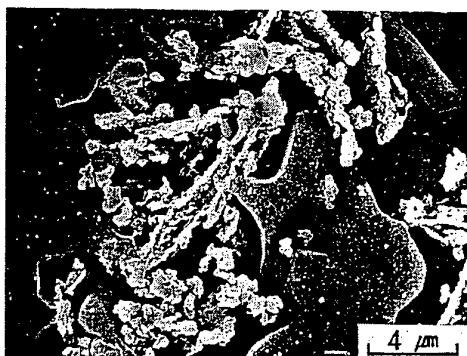
Figure 18. Particle morphologies in the Al-10wt.%Mg specimens after various reaction times: (a) 4 hours (b) 8 hours (c) 16 hours and (d) 32 hours.



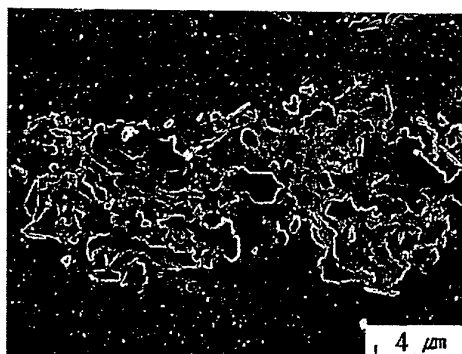
(a)



(b)



(c)



(d)

Figure 19. Particle morphologies in the Al-20wt.%Mg specimens after various reaction times: (a) 4 hours (b) 8 hours (c) 16 hours and (d) 32 hours.



(a)



(b)

Figure 20. Morphologies of the extracted particles from the Al-10wt.%Mg specimens with reaction times: (a) 16 hours and (b) 32 hours.



(a)



(b)

Figure 21. Morphologies of the extracted particles from the Al-20wt.%Mg specimens with reaction times: (a) 16 hours and (b) 32 hours.

The sizes of AlN particles were also found to be in the range from submicron to a few microns. In order to confirm that the extracted reaction products were AlN particles, the XRD analyses were carried out on the extracted particles. Figures 22 and 23 show the XRD patterns of the extracted particles obtained from the ingots that were prepared from the alloys initially containing 10 and 20 wt.% Mg, respectively. As can be seen in Figs. 22 and 23, most X-ray peaks were identified as the peaks corresponding to AlN.

In order to examine the thermal stability of the AlN particles from in the present *in-situ* nitridation process, solution heat treatments were carried out at 430°C for 24 hours on the ingots obtained by nitridation reaction. Figures 24 and 25 show the optical micrographs of the ingots that were obtained from the alloys initially containing 10 and 20 wt.% Mg, respectively, after solution heat treatment. The comparison of these micrographs with those shown in Figs. 11 and 12 obtained before solution heat treatment showed that there was little change in microstructure and morphologies of AlN particles as a result of solution heat treatment. These results indicated the reaction formed AlN particles are thermally stable.

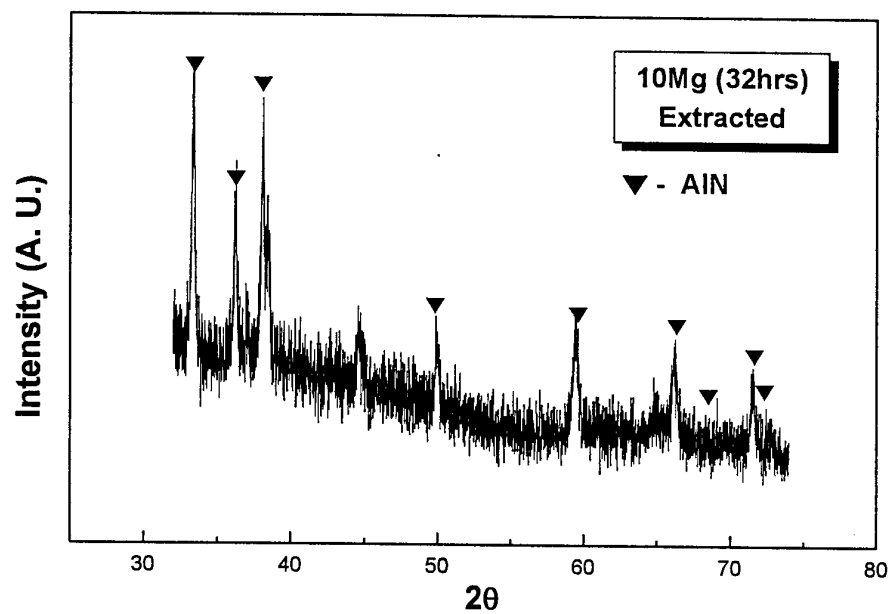


Figure 22. XRD pattern of the extracted particles from the Al-10wt.%Mg specimen after 32hrs reaction.

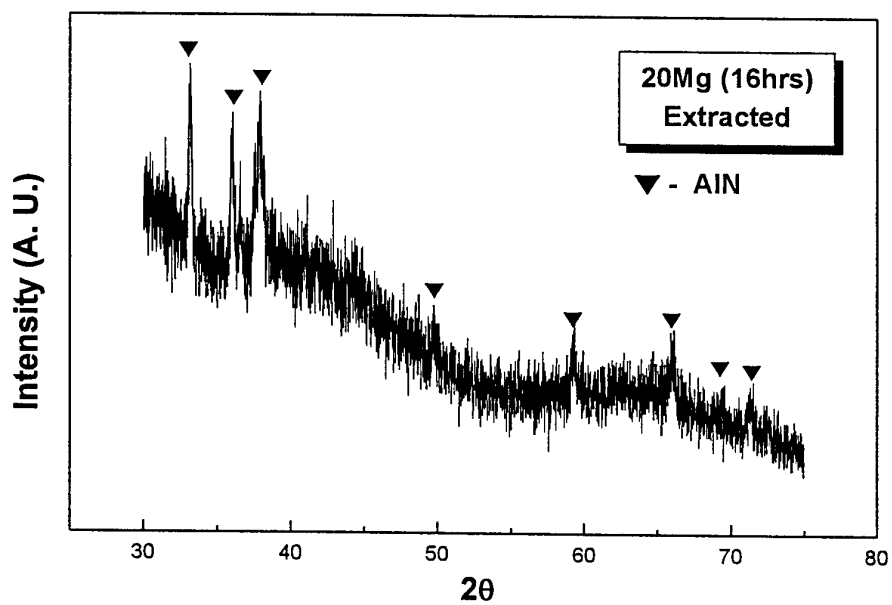
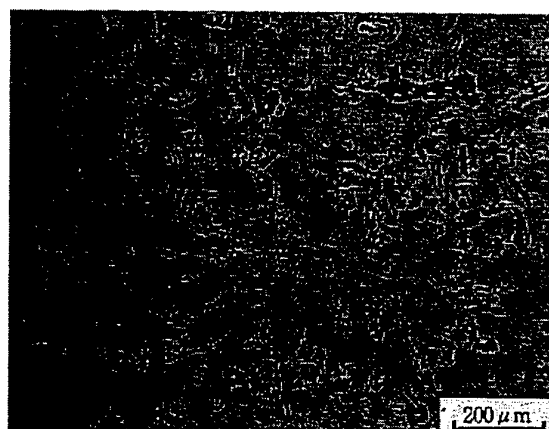
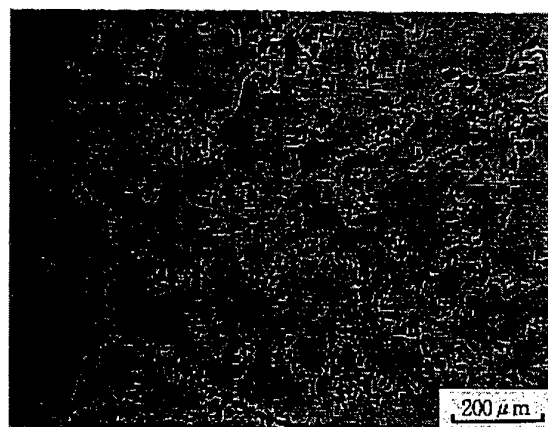


Figure 23. XRD pattern of the extracted particles from the Al-20wt.%Mg specimen after 16hrs reaction.

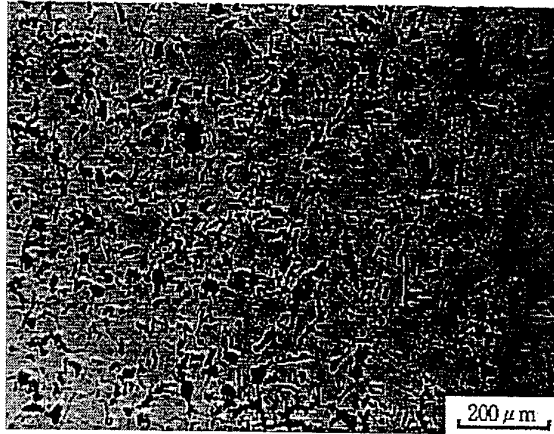


(a)

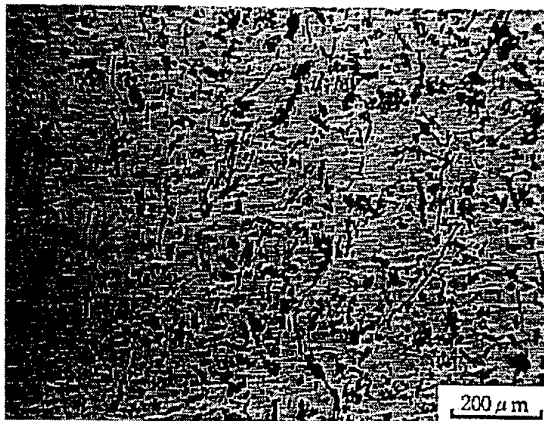


(b)

Figure 24. Optical micrographs of the solution heat treated Al-10wt.%Mg specimens:
(a) 16 hours and (b) 32 hours.



(a)



(b)

Figure 25. Optical micrographs of the solution heat treated Al-20wt.%Mg specimens:
(a) 16 hours and (b) 32 hours.

4-3. *In-situ* Nitridation of Al-5Si-10Mg Alloys

In many commercial Al alloys, Si is commonly added as an alloying element in order to improve the mechanical property and castability. It is, therefore, important to examine the effect of Si addition on nitridation reaction. In the present study, it was found that the fluidity of the melt decreased during the nitridation reaction of Al-Mg alloys because of the formation of AlN particles. Therefore, Si was added to the alloy melt in order to increase the fluidity of the melt in the present study. It has been reported, however, that the addition of Si suppresses the formation of AlN during the DIMOX process [10]. It is, therefore, important to examine the effect of Si addition on the *in-situ* nitridation reaction.

In order to examine the effect of Si on the nitridation reaction, 5 wt.% Si was added to the Al-10wt.%Mg alloy using the Al-25wt.%Si mother alloy. The resultant alloy had a similar chemical composition with the commercial 3xx alloy. The effect of Si addition was examined by the determination of the volume percent of AlN particles in the ingot after nitridation reaction at 900°C for various reaction times and the comparison of the results with those obtained from the Al-10wt.%Mg alloys. For the comparison of the results, all other experimental variables except the alloy composition were kept identical with those used for the Al-10wt.%Mg alloys. The reaction time was varied from 0 to 32 hours.

Figure 26 shows the optical micrographs obtained from the ingots after nitridation reaction of the alloys initially containing Al-5wt.%Si-10wt.%Mg alloys. The microstructure of the reacted ingots was quite different from that found in the ingots obtained from the Al-10wt.%Mg alloy. The Mg_2Si compound formed as layers in the microstructure of the ingots obtained after short reaction time. The amount of Mg_2Si compound appeared to decrease with increasing the reaction time. It was considered that the evaporation of Mg from the melt during the nitridation reaction at 900°C due to high vapor pressure decreased the Mg content in the melt and thus decreased the amount of Mg_2Si compound.

The XRD analyses were carried out of the powder samples obtained from the ingots after the nitridation reaction for various times. Figure 27 shows the XRD pattern of the specimen obtained from the ingot reacted for 32 hours. It could be clearly seen that the integral intensities of the X-ray peaks corresponding to AlN decreased significantly compared with those found in the ingots prepared from the Al-10wt.%Mg alloys, indicating the suppression of the nitridation reaction as a result of Si addition. The present result is in agreement with other reports on the effect of Si on the direct nitridation reaction.



(a)



(b)



(c)



(d)

Figure 26. Optical micrographs of the Al-5Si-10Mg specimens with reaction time: (a) 4 hours (b) 8 hours (c) 16 hours and (d) 32 hours.

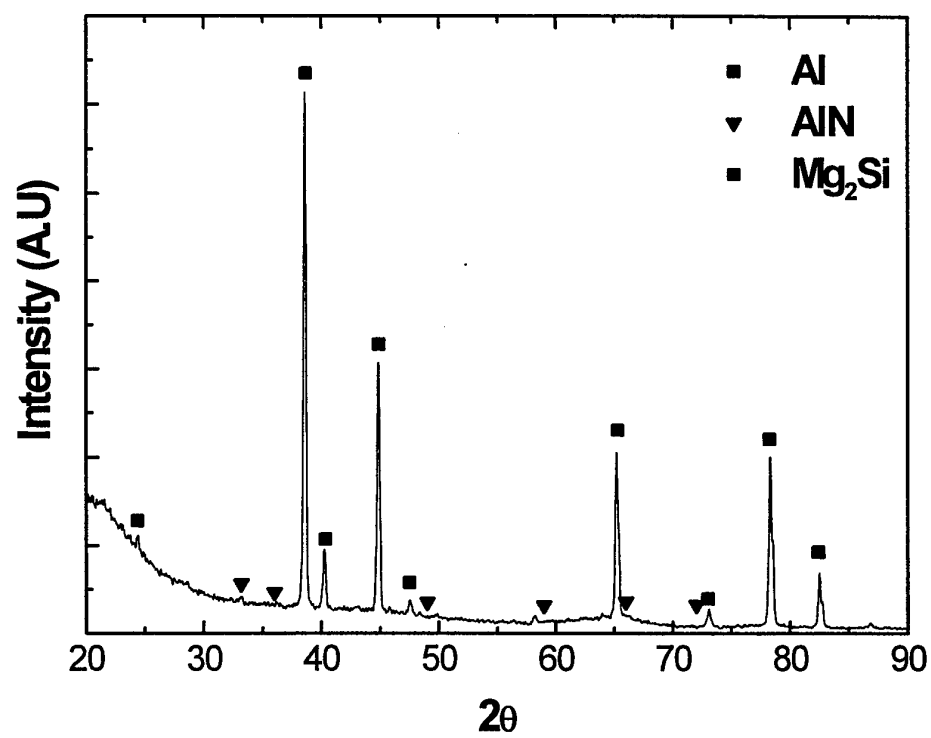


Figure 27. XRD pattern of the Al-5Si-10Mg specimen after 32 hrs reaction.

5. Conclusions

The primary objective of the present research is to provide a fundamental understanding of the processing science necessary to fabricate the AlN-reinforced Al composites via direct nitridation of the Al alloy melt. From the thermodynamic considerations, it was found that reduction of oxygen content below the critical level in the reaction gas was crucial for the successful formation of AlN without forming oxides. From the TGA results, it was shown that Mg could significantly increase the reaction rate between the Al melt and the reaction gas and promoted the formation of AlN. Based upon these experimental results, the reaction temperature for the *in-situ* nitridation experiments was chosen as 900. It was possible to produce a considerable amount of AlN particles in the Al matrix at a reaction temperature as low as 900 °C utilizing the *in-situ* nitridation reaction process developed in the present study by optimizing the processing variables including the content of alloying element, the composition and flow rate of the reaction gas, the reaction temperature and time, the stirring rate of the melt, etc. The optimum casting condition was also achieved for the uniform distribution of the reaction formed AlN particles in the matrix. The volume fraction of AlN was found to increase almost linearly with increasing the Mg content in the alloy and the reaction time. The shapes of AlN particles were found to be polygon, plate or rod-type from scanning electron microscopy of the extracted particles. The sizes of AlN particles were determined to be in the range from submicron to a few microns. In the case of Al-Si-Mg alloys, it was found that the integral intensities of the X-ray peaks corresponding to AlN decreased significantly after the nitridation reaction compared with those obtained from the Al-Mg alloys, indicating the suppression of the nitridation reaction by the Si addition. From the present study, it is concluded that the new innovative *in-situ* nitridation process developed in the present study can be successfully applied for the processing of high strength Al-AlN composites.

6. Further Research

In order to understand the exact mechanism of the nitridation reaction of Al melt and the effect of alloying element in the *in-situ* nitridation process, further research will be clearly required. Furthermore, the effects of various processing variables on the content and distribution of AlN particles need to be addressed in a detailed manner for the successful application of the present *in-situ* nitridation process for the production of high strength AlN reinforced Al composites in a commercially buyable way. The mechanical properties of the AlN reinforced Al composites should also be characterized and correlated to such microstructural variables as chemical composition, grain size, the content, size and distribution of reinforcements for the development of high strength composite materials.

Therefore, the following issues will be addressed in a detailed manner in the future research.

1. Identification of the nitridation mechanism in the *in-situ* reaction process.
2. Identification of the role of Mg in the *in-situ* reaction process.
3. Determination of the kinetics of the nitridation reaction.
4. Effect of other alloying elements including Si, Sr, Ca, etc.
5. Effect of reaction gas pressure.
6. Conditions for the reduction of reaction temperature and time.
7. Characterization of the mechanical properties of Al-AlN composites.
8. Relationship between mechanical properties and microstructural variables.

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