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PROCESSING OF IN-SITU AlMg/AlN METAL MATRIX COMPOSITES VIA REACTIVE GAS INJECTION

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Abstract: 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-Magnesium (AIMg) composites via Reactive Gas (N_2) Injection in the AIMg alloy melt. Aluminum nitride (AlN) matrix composites were prepared by a novel approach. It was possible to produce a considerable amount of AIN particles in the AI alloy matrix at a reaction temperature as low as 900 °C utilizing the "in-situ" nitration reaction process developed in the present study. The temperature of nitridation was found to play an important role in the formation of AlN particles. The volume fraction of AIN increases almost linearly with increasing the magnesium (Mg) content in the alloy and the reaction time. The shapes of AIN particles were found to have different forms, 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 AIMg/AIN composites. For particles and composite structure characterization some methods were used, including: light microscopy, scanning microscopy, quantitative analysis of selected composite regions, dilatometryand pn on disc friction. Composite structure and reinforcement distribution was compared with use of quantitative analysis. Morphology and diffraction pattern of aluminum nitride particles was shown. Typical structure of studied composites with microanalysis results was indicated. Aluminum nitride dispersion change was represented.

Key words: metal matrix composite, Reactive Gas Injection, aluminium alloys, "in-situ", nitride.

1. GENERAL INTRODUCTION

Aluminium nitride is a ceramic material with high thermal conductivity combined with high electrical resistance, low thermal expansion, high corrosion resistance and low density. Due to the unique combination of properties, AlN is attractive for refractory applications such as metal handling in semiconductor devices, heat sinks, electronic substrates, grinding media, seals, filler materials, etc. Also, enhanced mechanical performance, i.e. improved fracture toughness of AlN will allow new employment in structural applications (cutting tools). In addition to all the above applications, AlN is considered as one of the most effective materials for field emission device applications (Bahgat et al, 2006; Ye et al., 2005; Bello et al., 2012).

In previous investigations of the authors (Carcea 2008; Nedelcu et al., 2009), AlN synthesis and its growth mechanism through the reactive gas injection of nitrogen (RGI) gas have been studied and it was found that increasing the magnesium content an enhancement of the conversion level has been obtained. The reaction time, temperature and gas flow rate did not suffer any changes.

In the present paper, the authors has been applied the most favorable conditions obtained in their previous investigations for the synthesis of commercial AlN through RGI of AlMg/AlN composites obtained "in situ".

2. MANUFACTURING AND PROCESSING

Experimental procedure for obtaining composite materials such AlMg/AlN is based on the "in situ" technique and consists in introduction of reactive gas (nitrium) into the melt (AlMg alloy melt). Reinforcing particles (AlN) are formed from the reaction between molten metal and bubbling gas.

Table 1 presents the material and process parameters that have been tested in experiments on the formation of "in situ" AlN reinforcement particles.

3. EXPERIMENTAL PROCEDURE

The experiments have been carried out in an enclosed reaction chamber heated from outside by a vertical tube furnace (Fig. 1). The aluminum alloy (260 g, 99,9% pure Al and 99,9%pure Mg) has been introduced in an alumina crucible (3), which has been placed in a graphite larger crucible (2), inside a closed box melting chamber (1) made out of high grade stainless steel. The whole assembly has been introduced into the furnace (8) supported by an L-shape frame (7) which also holds the closed box supporting and adjustable pillar (4). The furnace has been provided from the bottom end cover and sealed at the top side by the chamber lid and the refractory material between the chamber and the furnace. The chamber lid has been water-cooled to room temperature and has been provided with several tubes for water and argon circulation.

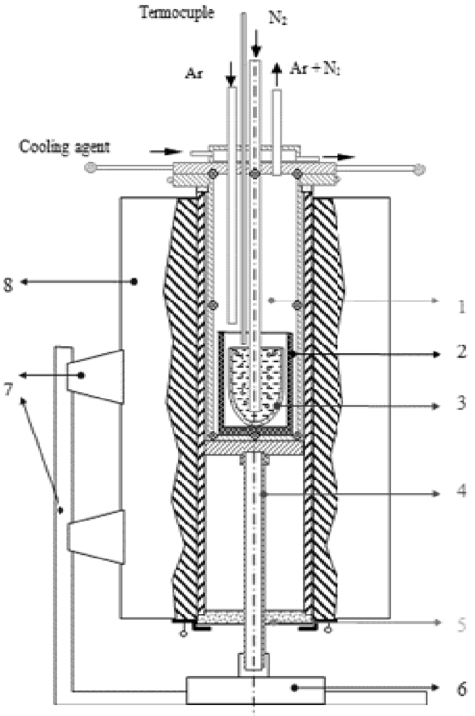


Fig. 1. Experimental installation (Soare et al., 2010)

4. RESULTS AND DISCUSSION

4.1. Friction wears of AlMg5/AlN and AlMg10/AlN composites obtainted “in situ”

The Universal Micro-Tribometer can be used effectively for the tribological testing of ferrous and non-ferrous metals, plastics, ceramics, paper, composites, thin and thick coatings, as well as of solid lubricants, lubricating fluids, oils and greases.

The UMT can accommodate both upper and lower samples of practically any shape. The upper specimen is connected to a vertical linear motion system that has a travel length of 150 mm. Wear measurements can be performed by the instrument to an accuracy of 50 nm. A precision spindle can rotate the lower specimen at speeds from 0.001-rpm up to 5,000-rpm. Ultra-accurate strain-gauge sensors perform simultaneous measurements of load and torque in two to six axes. The forces can be measured precisely in the ranges from milligrams to kilograms, with a resolution of 0.00003% of the full-scale and very high repeatability.

MMC type	Temp [ºC]	Mg [%gr]	Gas flow [l/min]	Bubbling time [min]
AlMg15/ AlN	1000	15	0,6	360
AlMg10/ AlN	1000	10	0,6	360
AlMg5/ AlN	1000	5	0,6	360

Table 1. Experimental parameters

A normal-load sensor provides feedback to the vertical motion controller, actively adjusting the sample position to ensure a constant load during testing. The tester has fully automated PC-based motor-control and data-acquisition, with a user-friendly proprietary software system in a Windows 2000/XP multitasking environment. The test data can be acquired, calculated and displayed in real time, as well as stored for future retrieval.

Figure 2 shows the basic Testing Unit without additional components. The drive motor for the carriage is hidden in the top. The lateral positioning system is mounted to the carriage and can be seen at the center of the picture.

The base of the Testing Unit has a fixture in the shape of a ring for mounting and leveling various drives for the lower test specimen.

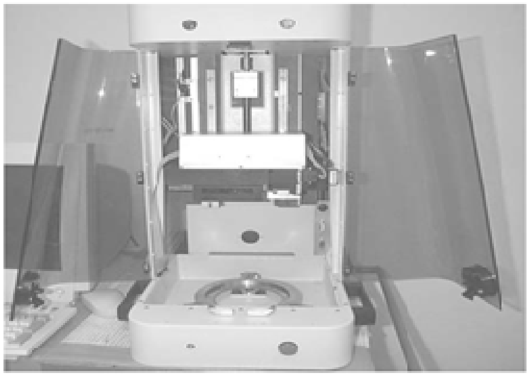


Fig. 2. Universal Micro-Tribometer

The friction wear coefficient of the AlMg5/AlN composite samples is showed in figure 3 and has the value equal with 0,94622.

For AlMg10/AlN composite obtained “in situ” via RGI the friction coefficient is presented in figure 4. In this case it has the value equal to 0,81428. It could be easily observed that there is a difference between AlMg5/AlN and AlMg10/AlN composites friction coefficient and, the difference is about 0,14.

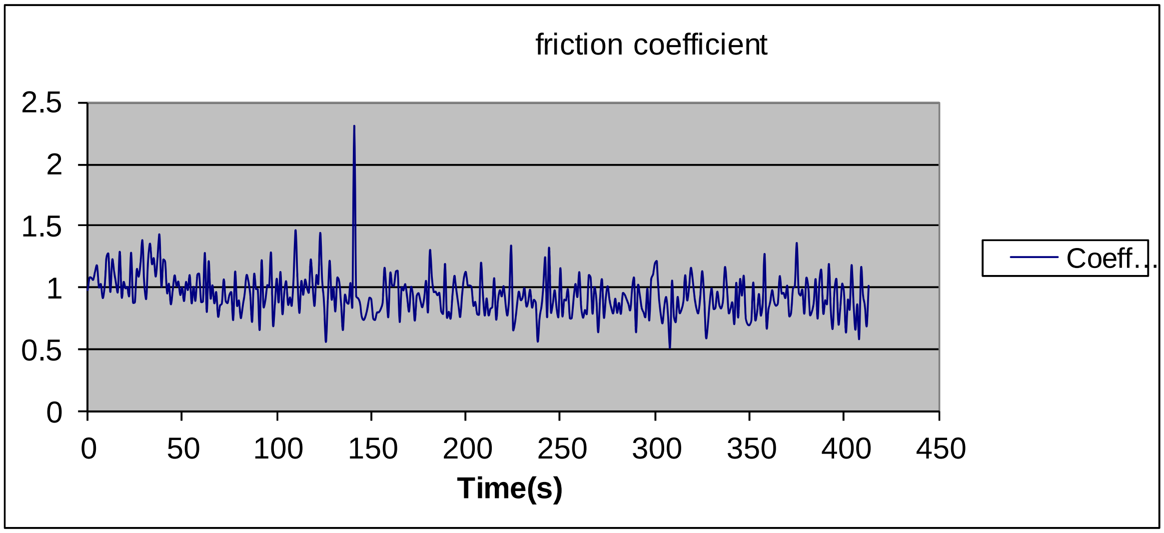


Fig. 3. Friction wear coefficient of the AlMg5/AlN composite, value equal with 0,94622

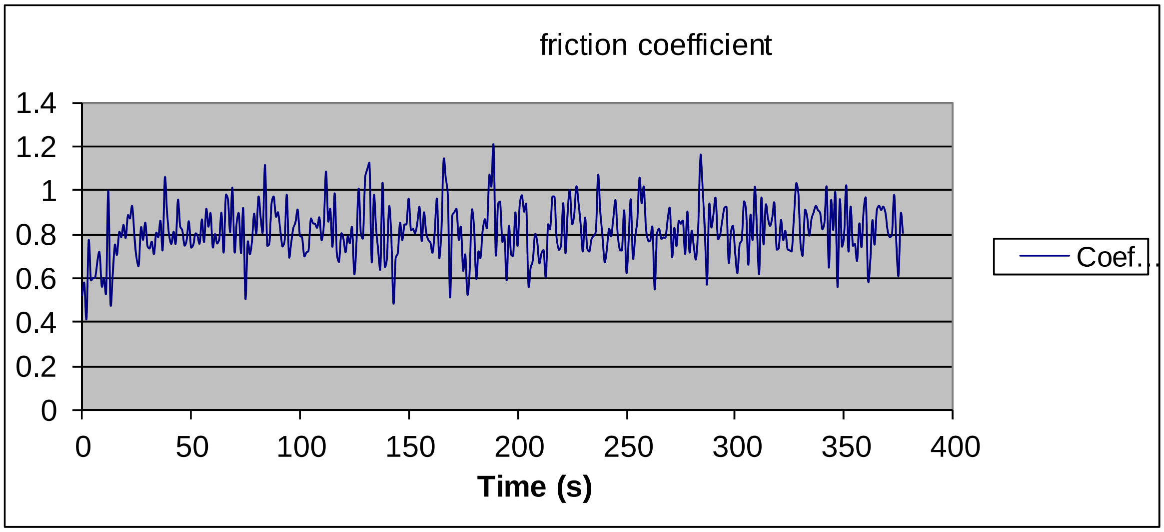


Fig. 4. Friction wear coefficient of the AlMg10/AlN composite, value equal with 0,81428

4.2. EDS analyses and chemical composition of AlMg/AlN composites

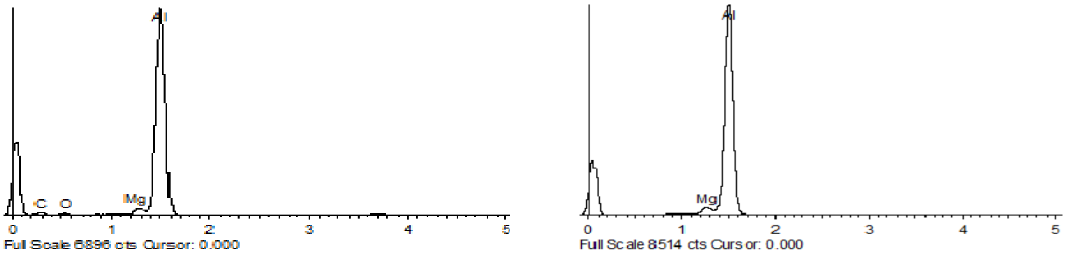
The composition of the isolated particles has been analyzed by EDS. The results revealed that these particles contained mainly Al, Mg, and N, along with a small amount of C, as shown below in tables for every composite in part.

Table 2. Chemical composition of AlMg5 matrix

Element	Weight%	Atomic%
Mg	3.24	3.58
Al	96.76	96.42
Totals	100.00	

Table 3. Chemical composition of AlMg5/AlN composite

Element	Weight%	Atomic%
C	9.30	13.65
N	4.95	6.23
O	53.33	58.77
Mg	2.11	1.53
Al	30.32	19.82
Totals	100.00	100.00



a) b)

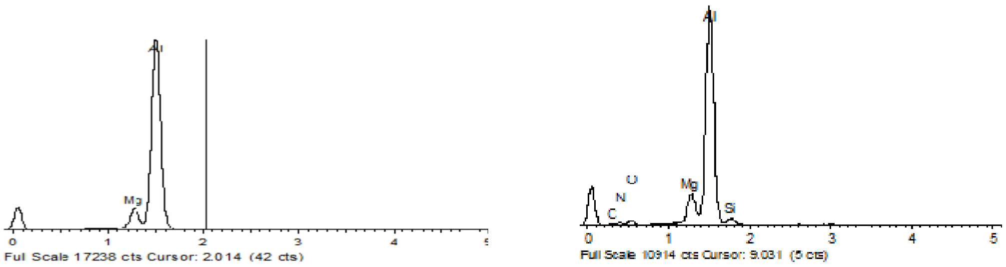
Fig. 5. EDS analyses of the composite AlMg5/AlN (microstructures made at laboratories of Politecnico di Torino, Department of Applied Science and Technology)
a) AlMg5 matrix without reinforcement particles
b) composite with AlMg5 matrix armed with AlN particles by gas infiltation

Table 4. Chemical composition of AlMg10 matrix

Element	Weight%	Atomic%
Mg	8.78	9.65
Al	91.22	90.35
Totals	100.00	100.00

Tabel 5. Chemical composition of AlMg10/AlN, point of interest: AlN

Element	Weight%	Atomic%
C	11.59	17.29
N	9.03	11.55
O	38.80	43.46
Mg	10.09	7.44
Al	30.48	20.25
Total	100.00	100.00



a) b)

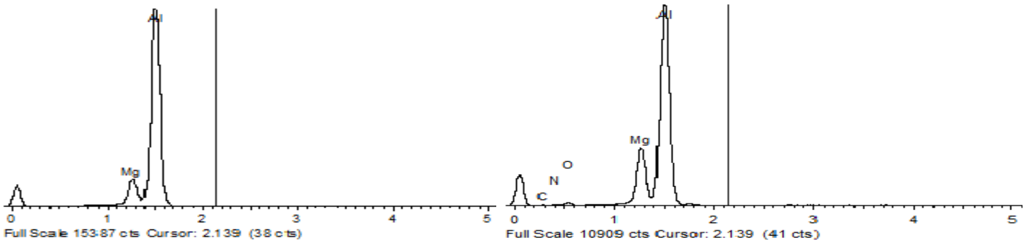
Fig. 6. EDS analyses of the composite AlMg10/AlN (microstructures made at laboratories of Politecnico di Torino, Department of Applied Science and Technology)
a) AlMg10 matrix without reinforcement particles
b) composite with AlMg10 matrix armed with AlN particles by gas infiltation

Table 6. Chemical composition of AlMg15 matrix

Element	Weight%	Atomic%
Mg	9.22	10.13
Al	90.78	89.87
Totals	100.00	

Tabel 7. Chemical composition of AlMg15/AlN, point of interest: AlN

Element	Weight%	Atomic%
N	8.61	14.85
O	3.95	5.97
Mg	9.65	9.58
Al	77.79	69.60
Totals	100.00	



a) b)

Fig. 7. EDS analyses of the composite AlMg15/AlN (microstructures made at laboratories of Politecnico di Torino, Department of Applied Science and Technology)
a) AlMg15 matrix without reinforcement particles
b) composite with AlMg15 matrix armed with AlN particles by gas infiltation

4. 3. Dilatometric examinations

Dilatometric examinations during heating at different temperatures for every kind of composite in part were performed by use of ultra high resolution Linseis L78 RITA (Rapid Induction Thermal Analysis). Te composite samples were slowly cooled in the crucible.

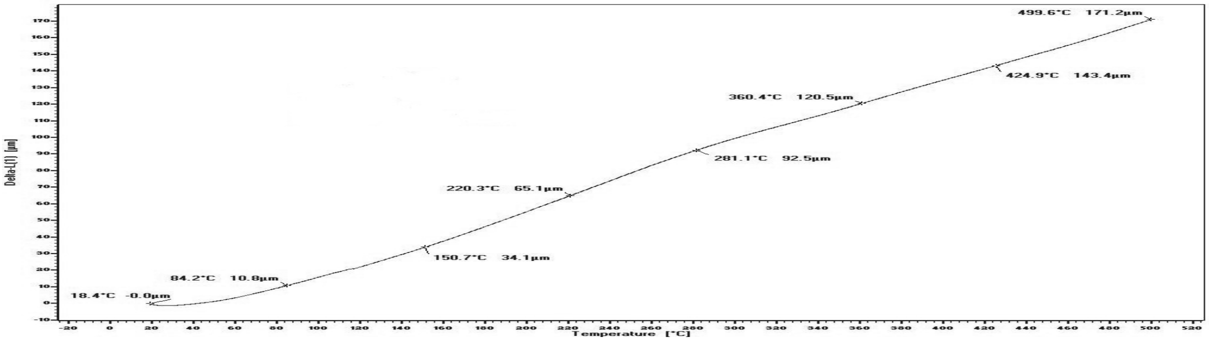


Fig. 8. The dilatometric curve of AlMg5/AlN composite after heating at 500°C for 50 min

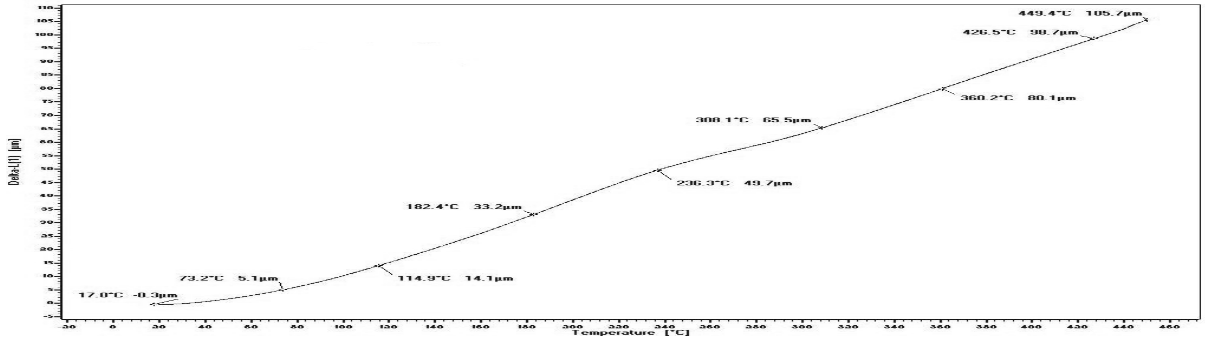


Fig. 9. The dilatometric curve of AlMg10/AlN composite after heating at 450°C for 45 min

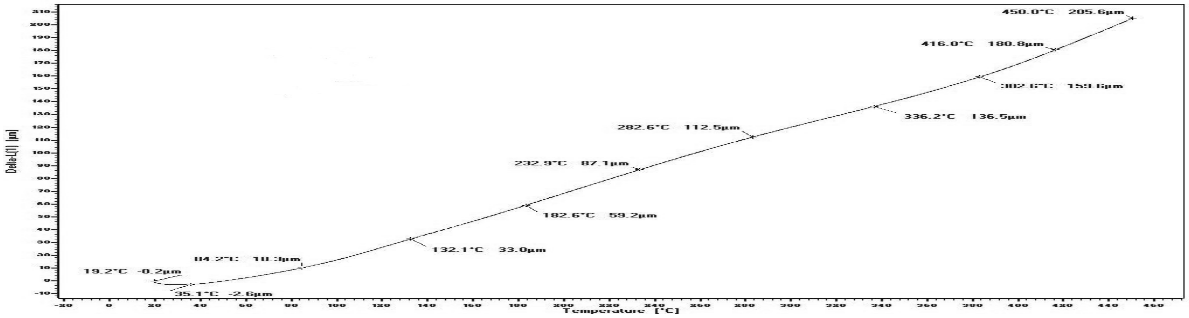


Fig. 10. The dilatometric curve of AlMg15/AlN composite after heating at 450°C for 45 min

4.4. AlMg/AlN composites microhardness

Vickers indentation method, standardized by STAS 492, has been used for mechanical characterization. The test consists of indenting the composite material with a diamond indenter using a force F with a quadratic pyramid square, diamond, perpendicular to test the surface and measuring the diagonal, d, of all residual print (Figure 11) (Dumitrache 2010).

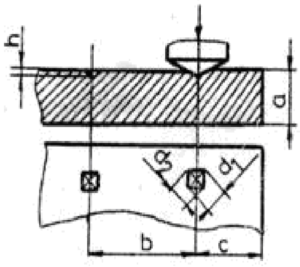


Fig. 11. Scheme of microhardness test after Vickers method.

The microhardness has been determined by a PMS 73 Microhardnes machine. Microhardness results are presented in table 8, 9 and 10 (table 8 for AlMg5/AlN composite, table 9 for AlMg10/AlN composite and table 10 for alMg15/AlN composite). There have been three attempts per sample, the penetration was performed using a pyramid penetrator type, pressing the sample and weight was 50g.

Vickers hardness (HV) for every three attempts was calculated using the equation (1);

$$HV = 20000 \cdot P/N^2 \quad [daN^2/mm^2] \qquad (1)$$

where: P = weight of pressing, N2 = number of divisions.

Table 8. Microhardness values for AlMg5/AlN

No. of attempts	P [g]	N [daN^2]	N^2 [daN^2]	HV [daN^2/mm^2]
1	100	175	30625	32.66
2	100	170	28900	34.61
3	100	168	28224	35.44
Average				34.24

Table 9. Microhardness values for AlMg10/AlN

No. of attempts	P [g]	N [daN^2]	N^2 [daN^2]	HV [daN^2/mm^2]
1	100	147	21609	46.28
2	100	144	20736	48.23
3	100	141	19881	50.30
Average				48.27

Table 10. Microhardness values for AlMg15/AlN

No. of attempts	P [g]	N [daN^2]	N^2 [daN^2]	HV [daN^2/mm^2]
1	100	118	13924	67.37
2	100	114	12996	76.95
3	100	112	12544	79.72
Average				74.68

5. CONCLUSIONS

Aluminium alloy matrix composites (AlMMC) are a group of materials more and more frequently used in modern engineering constructions because of their excellent properties, i.e. high specific elasticity modulus, high stiffness.

AlN particles were successfully grown via reaction gas injection of nitrium in melted AlMg matrix at 1000 °C.

It was found that Mg plays a key role in reducing the partial pressure of oxygen in the furnace chamber during the entire process favoring the development of AlN and suppress ing the formation of Al_2O_3.

Acknowledgement

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