IN SITU SYNTHESIS OF ALN IN AL–MG ALLOYS BY RGI

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Abstract: *This paper reports the results of an investigation on the in situ formation of AlN particles in an Al–Mg alloy through liquid nitridation. The AlN particles were produced in the Al–Mg melt by bubbling nitrogen gas through the molten alloy. Basic material concept, technology and some results of studies on aluminum matrix composite with dispersive aluminum nitride reinforcement was shown. Studied composites were manufactured with use of "in-situ" technique.*

Keywords: *Composite; AlN; Reactive Gas Injection; In situ particle formation; Casting.*

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 uses 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 [1÷3].

In related investigations of the authors, AlN synthesis and its growth mechanism through the reactive gas injection of nitrogen gas were studied [4, 5]. It was found that increasing the magnesium percentage enhanced the conversion extent. The reaction time, temperature and gas flow rate did not suffer changes.

In the present work, the authors applied the optimum condition from their previous investigations for the synthesis of commercial AlN through reactive gas injection of AlMg/AlN composites obtainted "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

Table 1: Experimental parameters

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

The experiments were carried out in an enclosed reaction chamber heated from outside by a vertical tube furnace (Figure 1). The aluminum alloy (260 g, 99,9% pure Al and 99,9%pure Mg) was introduced in an alumina crucible (3), which was placed in a graphite larger crucible (2), inside a closed box melting chamber (1) made out of high grade stainless steel. The entire assembly was 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 was provided with a 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 was water-cooled at room temperature and provided with several tubes for water and argon circulation.

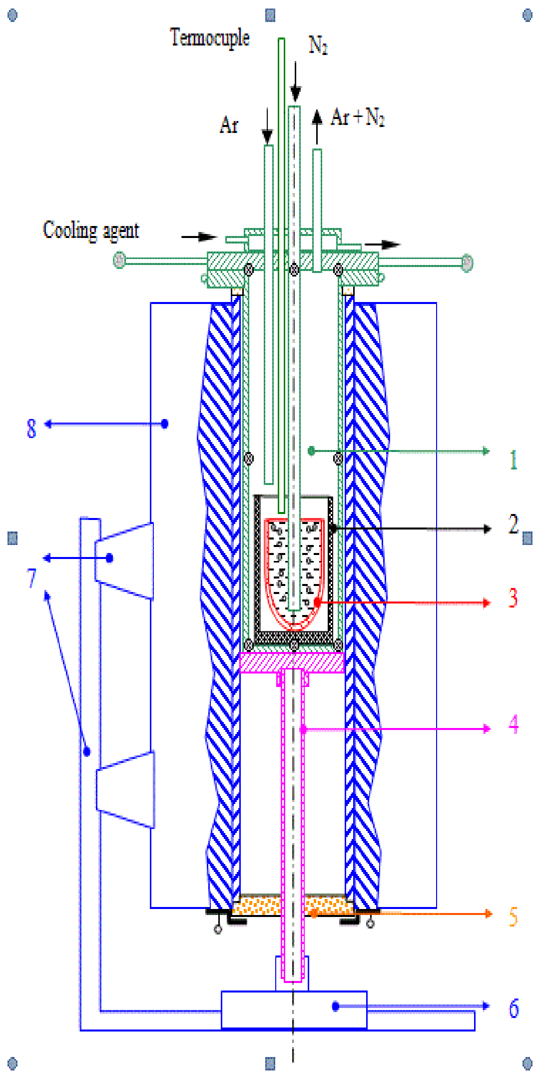


Figure1: Experimental installation [6]

4. Results and discussion

4.1. AlMg/AlN composites microhardness

Vickers method, standardized by STAS 492, consists in printing in a given time with a force F of a pervasive form of a quadratic pyramid square, diamond, perpendicular to test the surface and measuring the diagonal, d, of all residual print (Figure 2) [7].

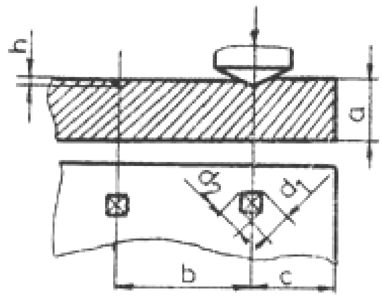


Figure 2: Scheme of microhardness test after Vickers method.

For determining microhardness was used a PMS 73 Microhardness machine. Microhardness results are presented in table 2, 3 and 4 (table 2 or AlMg5/AlN composite, table 3 for AlMg10/AlN composite and table 4 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 100g.

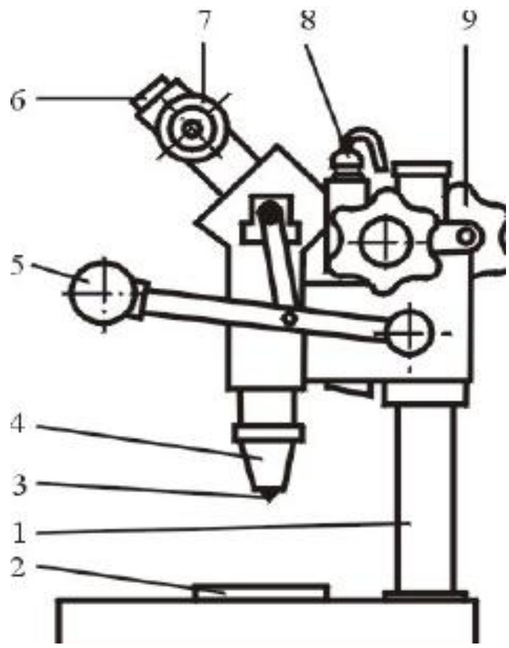


Figure 3: Microhardness machine used to determine the Vickers microhardness for AlMg/AlN omposite
1) support column; 2) motherboard; 3) penetrator; 4) protective cone; 5) lever; 6) eye; 7) ocular micrometer; 8) light; 9) hand handle [7]

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

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

where: P = weight of pressing, N^2 = number of divisions.

Table 2: 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	65.31
2	100	170	28900	69.21
3	100	168	28224	70.87
Average				68.47

Table 3: 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	92.56
2	100	144	20736	96.45
3	100	141	19881	100.60
Average				96.54

Table 4: 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	143.64
2	100	114	12996	153.89
3	100	112	12544	159.44
Average				152.32

4.2. 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.

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 4 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.

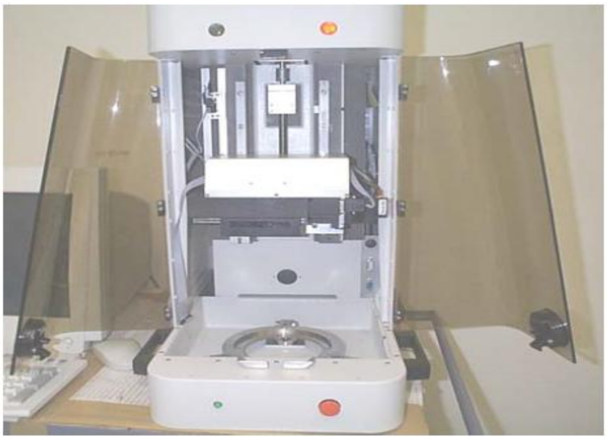


Figure 4: Universal Micro-Tribometer

The friction wear coefficient of the AlMg5/AlN composite samples is showed in figure 5 and has the value equal with 0,94622.

For AlMg10/AlN composite obtained "in situ" via RGI the fricyion coefficient is presented in figure 6. In this case it has the value equal with 0,81428. It could be easily seen the fact that there is a difference between AlMg5/AlN and AlMg10/AlN composites friction coefficient, the difference is around 0,14.

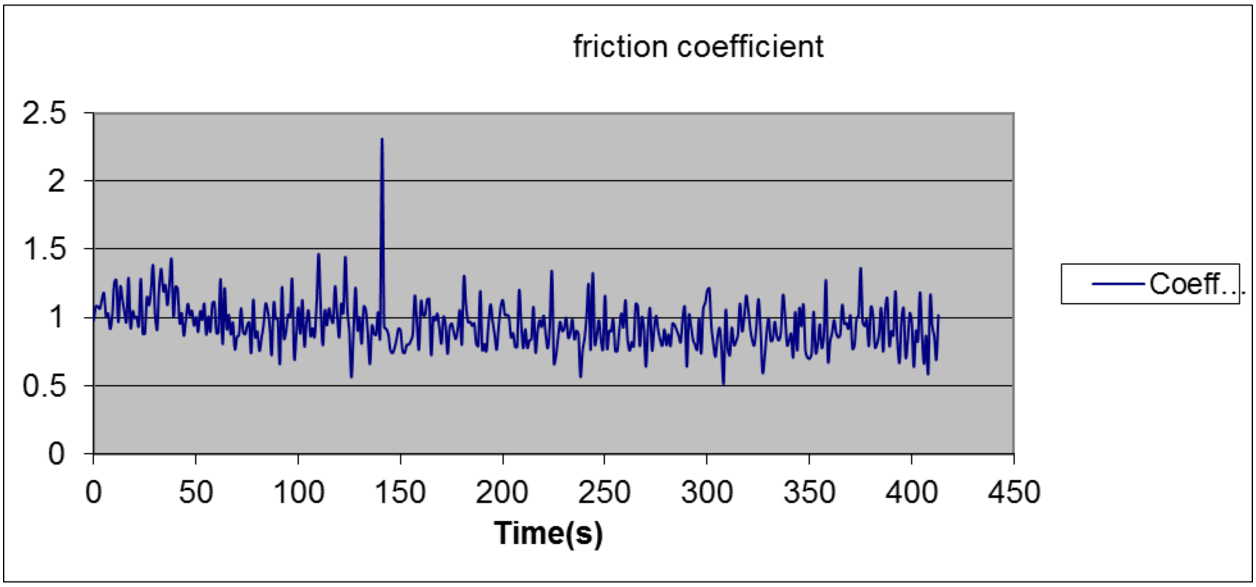


Figure 5: Friction wear coefficient of the AlMg5/AlN composite, value equal with 0,94622

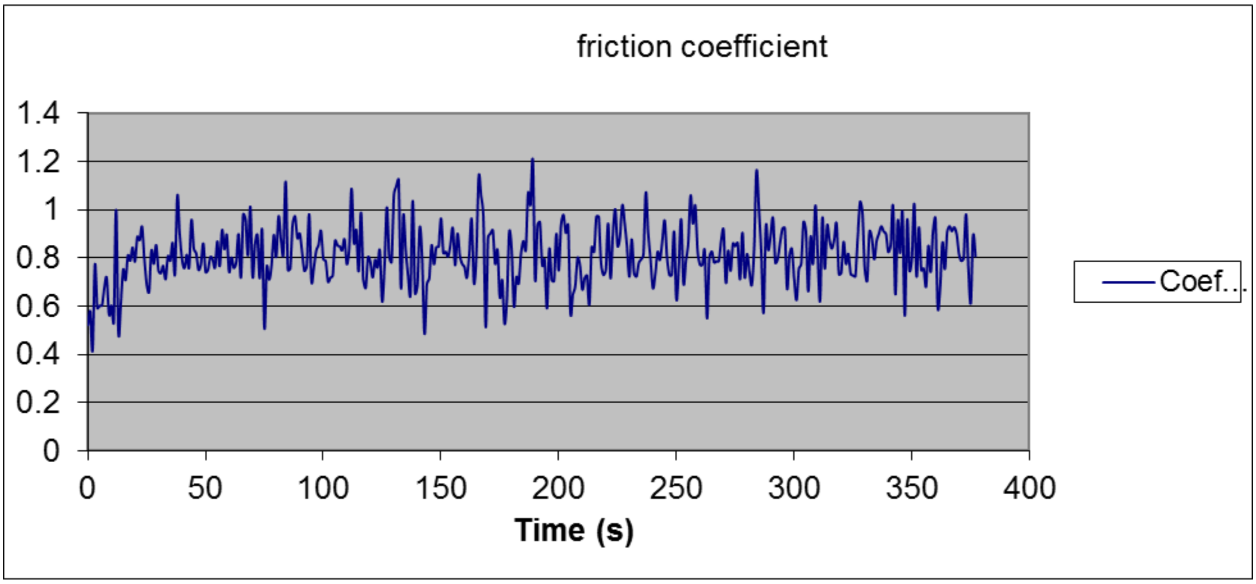


Figure 6: Friction wear coefficient of the AlMg10/AlN composite, value equal with 0,81428

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

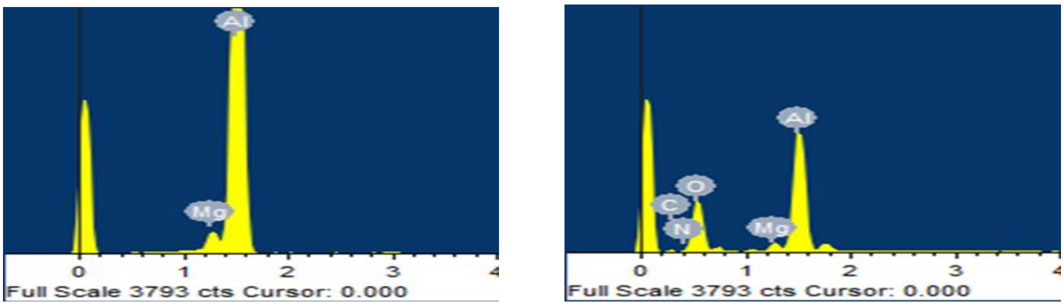
The composition of the isolated particles was analyzed by EDS. The results revealed that these particles contained mainly Al, Mg, and N, along with a small amount of C, as shown below in tables for every composite in part.

Table 5: Chemical composition of AlMg5 matrix

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

Table 6: 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)

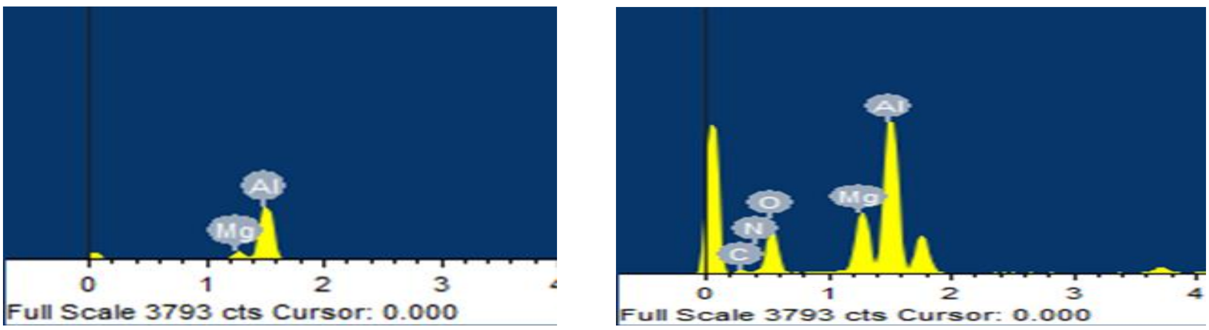
Figure 7: EDS analyses of the composite AlMg5/AlN (microstructures made at laboratories of Politecnico di Torino, Department of Applied Science and Technology)
a) AlMg5 matrix without reinforcement particles
b) composite with AlMg5 matrix armed with AlN particles by gas infiltation

Table 7: Chemical composition of AlMg10 matrix

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

Tabel 8: 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)

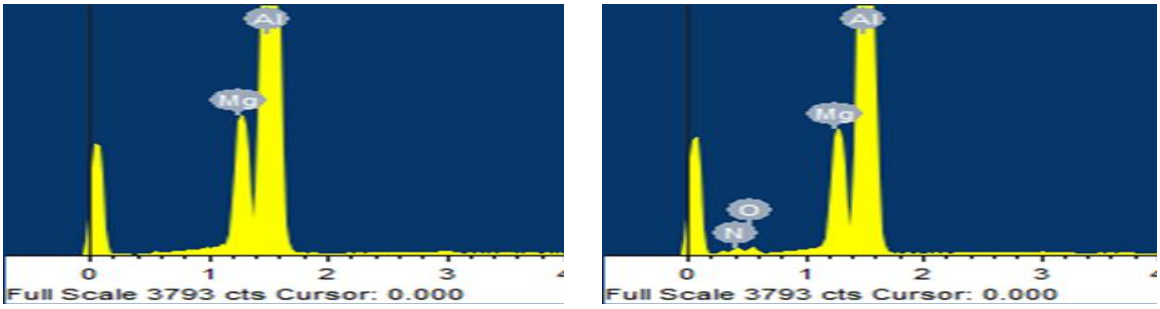
Figure 8: EDS analyses of the composite AlMg10/AlN (microstructures made at laboratories of Politecnico di Torino, Department of Applied Science and Technology)
a) AlMg10 matrix without reinforcement particles
b) composite with AlMg10 matrix armed with AlN particles by gas infiltation

Table 9: Chemical composition of AlMg15 matrix

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

Tabel 10: 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)

Figure 9: 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

5. Conclusions

Composites with an aluminium alloy matrix (AlMMC) are a group of materials which due to their properties (high specific elasticity modulus, high stiffness) are more and more frequently used in modern engineering constructions.

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

Mg plays a key role on reducing the partial pressure of oxygen in the furnace chamber during the entire process favouring the formation of AlN andsuppress ing the formation of Al_2O_3.

Acknowledgement

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