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Aluminium-Aluminium Nitride Composite - A Review of production methods.

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ABSTRACT

Aluminium-aluminium nitride (Al-AlN) composite has continued to attract more attention due to its wide area of application such as in the electronic industry, aerospace, high temperature applications, military and other areas. This review gives the recent advances in the processing methods of this composite with more emphasis on solid-gas and liquid-gas in-situ nitridation processes, which have attracted the interest most researchers as suggested by the available literatures. The paper also highlights the major challenges associated with nitridation of aluminium and possible means of addressing these challenges.

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INTRODUCTION

A composite material is made up of a base material usually referred to as the matrix and one or more other constituents usually referred to as the reinforcements (Colque & Grange, 1994), which are usually added to modify the behavior of the monolithic metals (Aigbodon & Hassan, 2007; Yang, Lan, & Li, 2004). The reinforcements are usually dispersed in the matrix with the objective of enhancing the properties of the matrix such as hardness, strength, wear resistance, corrosion resistance and other desired properties (Aigbodon & Hassan, 2007; Clyne, 2001; Fale, Likhite, & Bhatt, 2011). The recent research interest in the processing of composite has been centred on the use of light metals such as aluminium, magnesium and titanium, and others matrix materials such as polymer and inorganic substances that have been reinforced to produce materials with reduced weight but with enhanced mechanical and other properties that could be tailored to meet different applications (Lindroos & Talvitie, 1995). Composite materials have found applications in aerospace industries, military, automobiles, electronics, tooling industries and so on (Aigbodon & Hassan, 2007; Clyne, 2001).

Composite materials are classified based on (Fale *et al.*, 2011; Lindroos & Talvitie, 1995; Sharma, 2000): (i) the matrix whose purpose is to bind the reinforcement phases together; these include, Metal Matrix Composites (MMCs) in which the major proportion of the material (matrix) is a metallic element. Others are Polymer Matrices, Ceramic Matrices and Carbon/Graphite Matrices; (ii)

Based on the type of reinforcement phases used; these are classified into: particulates, fibres, whiskers, filled (particles and microspheres), flakes and directionally solidified eutectic/intermetallic reinforced composites (Lindroos & Talvitie, 1995; Sharma, 2000).

In this review, the advances in processing Al-AlN composite is being discussed with more emphasis on in-situ process of forming AlN in Al-matrix based on nitridation techniques.

1.1 General Methods of Processing Composites:

The methods of processing composites are classified into two: ex-situ process where the reinforcing phase is added into the matrix from an external source and in-situ process where the reinforcement is generated by the reaction with and/or in the matrix (Fale, Likhite, & Bhatt, 2014). Some of the main methods generally used in processing composites include: powder metallurgy route, stir casting, infiltration, deposition, plasma, self-propagating high temperature synthesis (SHS) (Chung, 2002; Fale *et al.*, 2011; Sharma, 2000). These processes could be solid-solid, solid-gas, liquid-solid or liquid-gas in nature. The mechanisms involved in the different methods have been grouped and discussed based on the methods used by different researchers from the oldest to the most recent research conducted as discussed in the next section.

2.0 Aluminium-aluminium nitride (Al-AlN) Composite:

AlN is a ceramic material characterized by high thermal conductivity, low dielectric constant, low coefficient of thermal expansivity that matches that

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of silicon, high melting point, large band gap, high electrical resistivity, impressive mechanical strength, wear and corrosion resistance and good resistance to thermal shock (Angappana, Jeneferb, Visuvasama, & Berchmansa, 2013; Kim *et al.*, 2015; Qiu & Gao, 2003; Smolen *et al.*, 2013). These attributes of AlN are of great interest due to the great potentials it has for various applications: The high melting temperature ($>2500^{\circ}\text{C}$) of AlN makes it thermally and chemically stable and suitable for application as a refractory material (Hou, Mutharasan, & Koczak, 1995). It's wide band gap makes it suitable for use in the electronic industry especially as semi-conductor material (Yamakawa, Tatami, Komeya, & Meguro, 2006). Other areas of applications of this material are: as heat sinks of mainframes in supercomputers, field emission devices and its potential for use in place of alumina (Angappana *et al.*, 2013; Moya *et al.*, 1997; Sheppard, 1990). There could be other areas of application of AlN not yet explored and I believe application of heat treatment of this material could bring out certain properties that could make them useful in other areas of applications not yet known.

The trends in forming Al-AlN composites have taken different dimensions. Different researchers have used different nitridation methods (i.e, methods of introduction of nitrogen) to produce Al-AlN composite both in the solid, liquid and gaseous states. The processing methods used to form Al-AlN

composite by different researchers in the last few decades are being presented.

2.1 Powder metallurgy route:

This is an example of ex-situ method whereby the matrix metal and reinforcement in the form of powder are mixed together, compacted and sintered at the required temperature to achieve the required density and other properties (Clyne, 2001; Lindroos & Talvitie, 1995; Sharma, 2000). The required aluminium nitride powder needed for this method has been produced in-situ by different researchers using different nitridation methods both in solid and liquid forms. Composites produced using these methods are faced with the problems of wetting, non-uniform distribution of the reinforcement in the matrix and difficulty to have material with equal density throughout, especially when articles with complex shapes or non-uniform cross-sections are to be produced (Fale *et al.*, 2011; Sharma, 2000).

2.1.1 Solid-gas nitridation of Al and its compounds:

Colque and Grange (Colque & Grange, 1994), proposed a new method of producing AlN using alumina (Al_2O_3) as the source of Al in 1994. In their study, alumina powder was placed at different positions inside a reactor while a mixture of NH_3 and N_2 containing 24% of NH_3 was passed through the chamber at 1200°C and 1350°C for different times ranging between 5 and 8 hours according to the arrangement in fig. (1).

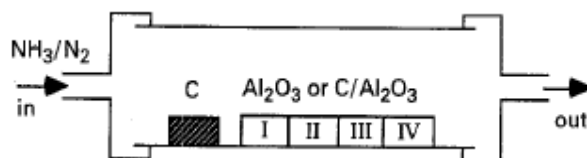
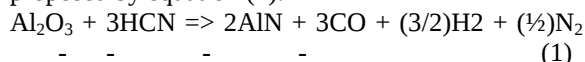


Fig. 1: A schematic representation of the reactor showing the position of C and Al_2O_3 **Source:** (Colque & Grange, 1994).

The results from XRD analysis showed that AlN produced was small in the absence of C and almost 100% in the presence of C. According to the authors, the formation of HCN in the latter would have probably facilitated the nitridation process as proposed by equation (1).

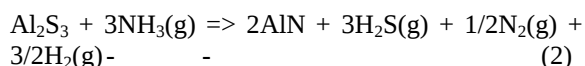


From the results, the authors observed that an increase in temperature and time help to increase the transformation of Al_2O_3 into AlN. The formation of cyanide during this process limits its application due to its toxic nature.

Al-AlN composite has also been synthesized by mechanofusion (i.e reaction being mechanically activated) of Al in the presence of NH_3 gas as a source of nitrogen (Calka & Nikolov, 1995). In this process, Al powder was milled by application of 600 kPa pressure on 25mm hardened steel balls that performed the milling and shearing of the powder for

200 hours giving rise to direct formation of AlN. XRD analysis showed Bragg's peaks indicating the presence of nano-composites consisting of AlN and Al. Using this method, the highest amount of AlN form was 88.28%. This method is promising considering the weight percent of Al converted to AlN. However, the authors concluded that this method is limited due to the possibility of cold-welding Al particle to form an Al layer on the ball mill hence hindering the process of forming AlN and the time taken to complete the process is too long.

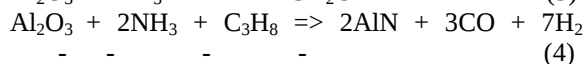
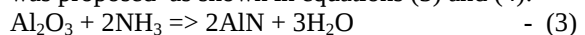
(Jung & Ahn, 2000), proposed a method of forming AlN by nitridation of aluminium sulphide (Al_2S_3) with NH_3 at lower temperature. In the study, Al_2S_3 was heated in a furnace at different temperatures ranging between 500°C and 1000°C for 5 hours in flowing ammonia. XRD analysis shows that AlN was formed at temperatures as low as 550°C after 5 hours. The reaction for the conversion is stated in equation (2) thus (Jung & Ahn, 2000):



Though the result established the formation of AlN in this study, the percentage conversion of Al_2S_3 to AlN was not established. Therefore, this method has to be investigated further before a recommendation can be made.

Wang, Wu, Tasi & Liu, (2000), prepared AlN by heating AlCl_3 in flowing mixture of 1:1 ratio NH_3 and N_2 . In the study, Al powder placed in a tube furnace was heated at different temperatures ranging from 600°C to 1150°C for 2 hours. XRD, TEM and SEM analyses revealed that at lower and higher temperatures, the morphology of AlN formed were amorphous and polyhedral structures respectively. From the plot of particle size against the reciprocal of temperature ($1/T$), the result shows that as the temperature increases, the particle size of the AlN produced reduces. Though the results showed that AlN was formed at temperature of 600°C and above, the percentage conversion from AlCl_3 to AlN was not established and the possibility of producing HCl during the process limits the application of this method (Wang, Wu, Tasi, & Liu, 2000),.

Al_2O_3 has also been transformed to AlN by gas-reduction method using ammonia (NH_3) and propane (C_3H_8) as reducing gases (Suehiro, Tatami, Meguro, Matsuo, & Komeya, 2002a). The process was carried out in alumina tube furnace under controlled atmosphere. The mechanism of formation of AlN was proposed as shown in equations (3) and (4):



XRD and SEM analyses revealed that AlN was formed during the process. The results showed that the presence of 0.5 vol % ammonia gas at 1500°C gave a maximum conversion of up to 94% within the first 30 minutes. It was reported that the highest conversion of Al to AlN observed was due to the thermodynamic influence of adding propane which also suppressed the polymorphic transformation of alumina. The authors concluded that this method was promising and could be used to produce AlN of any desired morphology by choosing alumina with the required morphology at the beginning of the process. However, the relatively high temperature involved in this process limits its potential for processing AlN due to high tendency of oxidation of Al and AlN and high cost of the furnace.

(Yamakawa *et al.*, 2006), achieved reduction-nitridation using a mixture of ammonia and propane gas similar with Suehiro, Tatami *et al.* 2002a, but their source of Al was $\text{Al}(\text{OH})_3$ rather than Al_2O_3 used in (Suehiro *et al.*, 2002a). SEM and XRD results obtained in this study revealed that highest amount of AlN obtained was at 1400°C which is 100°C lower than in (Suehiro *et al.*, 2002a). However, the percentage of $\text{Al}(\text{OH})_3$ converted to

AlN was not established to justify the use of this method.

Joo and Jung, (Joo & Jung, 2008b) investigated the influence of carbon monoxide on the carbothermal reduction and nitridation of Al_2O_3 using a mixture of N_2 and CO gases. In the study the precursor powder contain an alumina crucible was placed in a furnace in flowing ammonia at temperatures ranging between 800 and 1600°C . The result obtained from XRD patterns revealed the presence of AlN peaks when N_2 was used alone; but when N_2 was mixed with CO, both peaks of AlN and Al_2O_3 were observed. The peaks of Al_2O_3 were found to increase as both temperature and proportion of CO increase. The authors concluded that the presence CO retards nitridation process; therefore, the use of CO in this process is not recommended for nitridation of Al.

Agappana, Jeneferb *et al.* (Angappana *et al.*, 2013) proposed a method of producing AlN by direct nitridation using nitrogen gas in the presence and absence of additives. The additives employed in this study include NH_4Cl and urea. The operating temperature was varied between 700°C and 1000°C using a thermogravimetric analyzer (TGA). From the SEM and XRD analyses, the highest amount of AlN formed was 15% when NH_4Cl and urea were used at 900°C after 5 hours. According to the authors, the presence of NH_4Cl and urea did not significantly aid nitridation, hence, the ammonia gas obtained from decomposition of the additives were mostly used to produce NH_4N_3 whose composition was as high as 34%. Though this method was able to produce AlN at a relatively lower temperature, the conversion rate was too low and cannot be used as a viable means of producing AlN.

Other researchers who have contributed in this area of Solid-gas nitridation of Al are: (Lee, Kim, Lee, Kim, & Chun, 2013), (Ghosh Chaudhuri, Basu, Das, Mukherjee, & Mitra, 2013), (Jung, 2012), (YUE, TIAN, & CUI, 2011), (Kent, Drennan, & Schaffer, 2011), (D. Zhang, Liu, Cai, Liu, & Li, 2013), (Nasery, Pugh, & Medraj, 2011), just to mention a few.

2.1.2 Solid-solid Nitridation:

In this method, the initial raw materials are all in the solid states, though the nitridation may end up in a solid-gas or even liquid-gas as in (Fale, Likhite, & Bhatt, 2013). (Yamane, Shimada, & Disalvo, 1998), synthesized AlN using powder aluminium and sodium nitride as the source of nitrogen. The two materials were heated in a furnace at 600°C and 700°C for 12 and 24 hours. The results from SEM and XRD show that no AlN was formed at 600°C , but at 700°C more AlN was formed after 24 hours than after 12 hours. This process has the advantage of producing AlN at a lower temperature, but the length of time required to produce AlN limits its application. (Abbasi, Shariat, & Javadpour, 2013),

successfully produced AlN in 2013 using Al powder, silicon nitride and carbon all in solid states. The mechanism of the process is presented according to equation (8).



Fewer number available literatures in solid-solid nitridation suggests that not much has been done in the area.

2.2 Liquid-gas in-situ Nitridation of Aluminium:

In this method, nitrogen containing gases are introduced directly into molten Al to form Al-AlN in situ. (Hou *et al.*, 1995), studied the feasibility of nitriding molten aluminium alloys at temperatures ranging from 700 to 1500°C using nitrogen and ammonia gases as sources of nitrogen and held for time ranging from 1 to 60 hours. A mixture Mg and Si were added at different ratios to aid direct and indirect nitridation. The setting for the research is as shown figure 2

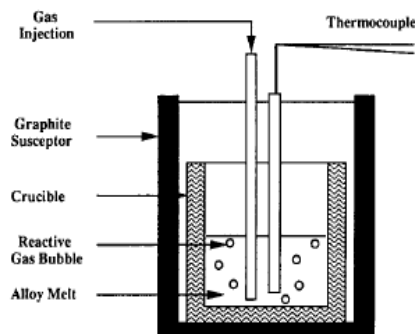


Fig. 2: Schematic reactor geometry **Source:** Hou *et al.* (1995).

From the study, the authors established that Mg plays two roles: as a catalyst and as an indirect source of nitrogen according to equations (5) and (6):

$$3\text{Mg} + \text{N}_2 \Rightarrow \text{Mg}_3\text{N}_2 \quad (5)$$

$$2\text{Al} + \text{Mg}_3\text{N}_2 \Rightarrow 2\text{AlN} + 3\text{Mg} \quad (6)$$

XRD analysis shows that the highest amount of AlN formed was 17 wt% at a temperature of above 1000°C. The authors concluded that AlN could be produced using ammonia and nitrogen gases in the presence of Mg and Si and that there is no significant difference in the use of either of ammonia or nitrogen gas. The major drawbacks associated with this process are: (i) the AlN was only formed on the surface of the ingot rather than being evenly distributed in Al matrix; (ii) percentage conversion of Al to AlN was too low (17%) as compared to the solid-gas method and (iii) high temperature involved in the process. This method need to be modified to improve the amount of AlN formed for it to be viable.

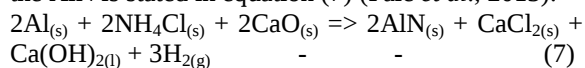
(Zheng & Reddy, 2004), investigated the mechanism of the in-situ method of processing Al-AlN by nitrogen bubbling into liquid aluminium. The research was conducted at temperatures ranging from 1173K – 1573K inside a resistance tube furnace. XRD analyses of the produced samples show that there were no significant formation of AlN when N₂ was used in its as-received form even at temperature of 1573K. However, when deoxidized nitrogen was used, XRD analysis shows peaks of AlN, which were only formed on the top portion of the Al melt. EDS quantified the content of Al and N₂ to be about 51.8% and 48.2% respectively. The authors reported

that it is possible to form AlN by bubbling nitrogen into Al melt when the pressure of oxygen is below a certain threshold value. In conclusion, it was reported that the highest amount of AlN formed in 80g of Al melt was 7.38g, which was also not evenly distributed in Al matrix as reported by Hou Mutharasan *et al* (Hou *et al.*, 1995). With the small amount of AlN formed compared to what is obtainable in solid-gas nitridation, this method cannot be recommended for processing Al-AlN unless the method is improved upon.

Al-AlN has been produced and synthesized in situ and characterized (Kumari, Pillai, & Pai, 2011), using nitrogen gas bubbling technique at temperature of 900°C for the time ranging between 2 and 4 hours and gas flow rate between 0.4 to 0.6 litre/minute. The furnace setting was same as the one proposed by Hou Mutharasan *et al.* (Hou *et al.*, 1995). From the XRD, SEM and TEM analyses, the results show that the AlN formed was more evenly distributed than in (Hou *et al.*, 1995) and (Zheng & Reddy, 2004). The results also show that the amount of AlN formed in Al matrix and the hardness of the produced composites increased with amount of Mg, N₂ flow rate and time of bubbling. However, the authors reported that there was an agglomeration of AlN particles which was still a major challenge to be corrected.

(Fale *et al.*, 2013), investigated the factors that influence the nucleation of AlN in aluminium matrix by nitridation. In the study, ammonium chloride and calcium oxide were used as the nitridation precursor at temperatures of 700°C, 800°C and 900°C in a Muffle furnace. XRD and SEM analyses show that

AlN was formed. The equation for the formation of the AlN is stated in equation (7) (Fale *et al.*, 2013):



The authors reported that higher temperatures increase the critical radius for the formation of AlN hence hindering nucleation. It was also stated that higher temperatures increase the fluidity of the melt hence making it easier for N₂ gas formed inside the melt to escape faster, hence reducing the contact period between N₂ and Al; consequently reducing the tendency for nucleation. The authors also observed that lower temperatures favour formation of smaller grain sizes of AlN hence enhancing the hardness and strength of the produced composites. This method is limited due to the lower wt% of AlN formed and due to the higher tendency for agglomeration as a result of its high surface energy as proposed by (Kumari *et al.*, 2011).

Other researchers that have worked in this area include: (Daniel & Murthy, 1995), (Yan, Chen, Jiang, & Wu, 2009), Roskosz, (Roskosz, Bouhifd, Jephcoat, Marty, & Mysen, 2013), (Peter, Florea, Bălătescu, & Carcea, 2014), just to mention a few. The major limitations in liquid-gas nitridation are the low conversion of Al to AlN, agglomeration of AlN particles and difficulty in stirring during the nitridation process. There is need to improve this method to boost its potential use considering the advantages attributed to liquid-gas nitridation method.

2.3 Stir Casting Method:

This method is carried out by first melting the metal above its melting point, then followed by the addition of a reinforcement in the form of solid particles (already produced using other means) then stirring to ensure more evenly distribution of the hard phase in the matrix. Researchers in this area include: (Hashim, Looney, & Hashmi, 1999), (Prabu, Karunamoorthy, Kathiresan, & Mohan, 2006), (Chatterjee, Sinha, Das, Ghosh, & Basumallick, 2013); just to mention a few. The major challenges associated with this method include: the possibility of introduction of impurity (Chatterjee *et al.*, 2013; Fale *et al.*, 2011) associated with production method or environment of the reinforcing phases, poor distribution of the reinforcement in the matrix during mixing due to insufficient time of stirring while maintaining the metal in a liquid state, which often result in agglomeration and structural inhomogeneity (Prabu *et al.*, 2006) and problem of insufficient wetting (Dolatkhah, Golbabaei, Besharati Givi, & Molaiekiya, 2012; Kim *et al.*, 2015) of the reinforcement by the matrix, hence reducing the cohesion between the matrix and the reinforcement and the mechanical properties of the produced composites (Chatterjee *et al.*, 2013; Dolatkhah *et al.*, 2012; Hull, 1996).

2.4 Infiltration method:

This method is used to inject molten metal into a porous or loosely preformed powder/short fibre, which has been lightly sintered so that pores can be eliminated to give a material high density. It is driven by capillary action (Sercombe & Schaffer, 2004). This method is also used to apply the matrix metal on an already pre-formed continuous fibre to complete the fabrication process (Hull, 1996; Sharma, 2000). The process is carried out through reactive metal infiltration or chemical vapour infiltration techniques (Zhao, Gao, & Jin, 2006). The process proffers solution to the dimensional problems associated with non-uniform densification or shrinkage of powder compacts during the powder metallurgy process or when pressing materials with non-uniform cross-section or complex shapes (Li, Sun, Imai, Mimoto, & Kondoh, 2013; Norgren, García, Blomqvist, & Yin, 2015; Sercombe & Schaffer, 2004). The process is enhanced by the reduction of grain size to the size just above the sub-micron level.

2.5 Self-propagating High-Temperature Synthesis (SHS):

This method has been used to produce Al-AlN based on exothermic reaction process (Nikhelesh Chawla and Chawla, 1998). Here, once the reaction starts, it can be sustained due to its exothermic nature, such that single crystals grow to form long fibres. In addition to nitrides of different metals and non-metals, SHS has been used to produce carbides, salicides, hydrides, carbonites, cemented carbides, intermetallics, etc. (Mossino, 2004). SHS has been carried out in the presence of other ammonium salts as additive such as NH₄Cl (Rosenband & Gany, 2007) and NH₄F (Fu, Chen, Xu, & Ferreira, 2005). These salts produce HCl and HF respectively that tend to react with Al hence dissolving the protective barrier and promoting AlN formation. The parameters that affect SHS include: grain size, nature of green compact, ignition, stoichiometry of the reactants, evolution of gas species and the effect of gas pressure (Fu *et al.*, 2005; Mossino, 2004). Aluminium nitride has also been produced by self-propagating combustion synthesis as reported by Ye and Liu (Yeh & Liu, 2007) and Kexin, Chen *et al.* (Kexin, Haibo, Heping, & Ferreira, 2000) just to mention a few. This process is limited by high temperature and pressure required to start the process and low yield of AlN.

2.6 Deposition Processes:

This could be in the form of physical or chemical vapour deposition (Dollet, Casaux, Chaix, & Dupuy, 2002). In physical vapour deposition (PVD), a material is heated and taken into the vapour phase, while in chemical vapour deposition (CVD), heating or chemical reaction occurs to produce the vapour, which in both cases, finally condense and are deposited on a preformed material to complete the

fabrication process by formation of continuous fibre composites (Chung, 2002; Hull, 1996). Other deposition methods include; electrodeposition, spray deposition (in which a molten metal is produced and atomized using a stream of fluid) and immersion plating (Lindroos & Talvitie, 1995). Though vapour deposition processes have the potentials for certain special fabrications and can guarantee high purity composite materials, the process is time consuming and cannot facilitate mass production. This area has only attracted few researchers for production of AlN such as: Dollet *et al.* (Dollet *et al.*, 2002), (Bashirzadeh, Azarmi, Leither, & Karami, 2013) and (De Sanctis, Valentini, & Solina, 1997), etc.

2.7 Plasma-assisted Nitridation:

This process is primarily used as a surface treatment method to impart wear resistance and enhance surface quality rather than a means of producing AlN for other purposes (Stock, Jarms, Seidel, & Döring, 1997). The mechanism of this process involves three stages (Quast, Mayr, & Stock, 1999): (i) formation of an oxide film on the surface of the material, (ii) sputtering process which is carried out as a means of dissolving the oxide film and cleaning the surface of the material and (iii) nitriding process that forms AlN on the surface of aluminium. The process is divided into arc assisted and ion beam techniques which are the means of producing the mass and energy required to hit the cathode during the pre-sputtering processes (Quast, Mayr, Stock, Podlesak, & Wielage, 2001). The followings are the parameters that affect the plasma process (Stock *et al.*, 1997): ion energy, ion mass, target temperature, incidence angle and presence of alloying elements. Some of the researchers in this area include: (Ageorges *et al.*, 1991), (Shi, Chen, Wang, Qiao, & Jin, 2011) (F. Y. Zhang & Yan, 2014), etc. This process is only limited to the enhancement of surfaces and cannot be used to produce AlN required for other fabrication purposes.

3.0 Conclusion:

All the processes discussed in this review have been used to produce AlN at various degrees of conversion and for different purposes. The major challenges generally identified by the authors during nitridation, depending on the method used, include: problem of wetting which causes de-bonding of the composites hence causing failure of the material during service; problem of oxidation of Al to form Al_2O_3 which is the major challenge during the nitridation process, difficulty for nitrogen gas to penetrate a large mass of aluminium powder as in solid-gas nitridation, formation of protective layer of AlN that tends to hinder the growth of nitride and the high temperature involves which makes it more difficult to check oxidation of Al and also increases the initial cost of procurement of equipment.

However, of all the methods used to produce AlN, Solid-gas nitridation has given the highest conversion of Al to AlN (94%), (Suehiro *et al.*, 2002a). The AlN produced using a solid-gas method is usually an intermediate stage of production and may require further processing to produce the finished products mostly by powder metallurgy route or stir casting techniques. This tends to increase the cost of production of the final products, introduce impurities during processing or handling of the powder and may bring about the problem of wetting in the finished products. In view of these problems associated with solid-gas nitridation and ex situ method of processing composites, I recommend the use of liquid-gas nitridation for production of Al-AlN composite due to the benefits associated with the process, especially the possibility of producing clean AlN reinforcement in an Al-matrix for use in the electronic industry. Liquid gas nitridation also reduces the problem of wetting and of course, cuts down the stages involved in producing the composite hence reducing the cost of production. Though percentage conversion of Al to AlN has been very low (maximum 34%) according to the literatures, there is a need for improvement to increase the potential use of direct (liquid) nitridation method.

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