Review on the Vapour-Phase Synthesis of Aluminum Nitride Powder Using Thermal Plasmas†

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Abstract

Production of high purity aluminum nitride powder which can be processed into electronic packages at a cost competitive with existing lower performance ceramic packages has promoted the development of many different synthesis methods. A survey of the synthesis of ultrafine AlN powders using thermal plasmas is presented. Laboratory scale experiments, applying inductively coupled and transferred-arc plasma systems, have been used to produce nanosize powders by vapour-phase reaction between aluminum vapour and ammonia, using a reduced number of steps. The specific surface area of the powders reported are in the 12 to 280 m^2/g range. Sinterability experiments have demonstrated that these powders can be compacted to full density at lower temperatures than powders produced by conventional methods.

1. Introduction

A noticeable progress has been made in the past few decades in the field of ceramic materials, based mainly on a better understanding of microstructure and its development during processing. Because of the key role that the starting material plays in the determination of the properties of the finished ceramic, this progress has been followed by a growing interest in the production of high quality ceramic powders [1], [2]. Many innovative powder synthesis methods have been proposed, addressing the fulfillment of requirements such as purity, particle size, particle size distribution, and particle morphology. One such method uses thermal plasma as the processing medium. The application of this technique to the production of powders of many different compositions has been demonstrated at the laboratory scale since the early 60's. Its most explored feature in these applications is the ability to carry out high temperature vapour phase reactions, which are followed by a fast quenching resulting in the formation of finely dispersed powders. Other highly valued features associated with the plasma synthesis are the reduced number of processing steps, and a clean processing environment that contributes to the production of high purity materials.

The application of thermal plasmas to the synthesis of refractory ceramic powders, which invariably require high temperature processing, is of particular interest. Ceramic powders such as oxides, carbides and nitrides have been successfully synthesized using this technique. Literature reviews on this subject were carried out by Hamblyn [3] (1975), Fauchais et al. [4] (1983), Akashi [5] (1987), and Kong and Lau [6] (1990). The plasma synthesis of aluminum nitride (AlN) has attracted a great deal of attention. Its current and future potential were reviewed by Sheppard [2] (1989), Mroz [7] (1993), and Knudsen [8] (1995). AlN synthesis methods were reviewed by Selvaduray and Sheet [9] (1993). AlN is a covalent ceramic that has a high thermal conductivity and good dielectric properties. It has a thermal expansion coefficient closely matching that of silicon. This unique combination of properties makes AlN especially useful as a substrate material for electronic packagings. Compared with conventional alumina (Al_2O_3) substrates, the high thermal conductivity of AlN provides a higher energy dissipation that allows the development of electronic packages with higher density of components. Because AlN exhibits high strength and high temperature stability associated with most non-oxide materials, a

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variety of other applications also exist for AlN as structural ceramic [7]. Armour for aircraft and vehicles are currently the predominant structural application. High temperature applications involve AlN crucibles and hardware to process a wide variety of molten metals and salts. In metals like aluminum, AlN can be used to strengthen the matrix. In polymers, it can be used to increase stiffness, reduce the thermal expansion, or increase the thermal conductivity. Also, optical applications exist for AlN since it can be made translucent [10], or it can be added to alumina (Al_2O_3) to produce transparency in visible light [11].

It is recognized that AlN powder synthesis as well as its sintering procedure still need to be technologically and economically improved before these ceramics can be used to their full capability [7]. AlN powers are currently produced by two main processes: (a) the carbothermal reduction of alumina in the presence of nitrogen (N_2); and (b) the direct nitridation of Al powder. The major barriers to the large scale use of AlN are: the high cost of AlN powders [8]; and due to the presence of impurities, producing high thermal conductivity AlN is a major technological challenge [12]. Oxygen in the AlN lattice is considered the principal impurity responsible for lowering its thermal conductivity [7], [12].

Notwithstanding the large amount of research work in this field, scaling up of plasma reactors from the few grams/hr of lab scale experiments to the rates needed for industrial scale production is still a major problem. A technological barrier encountered in these developments is the limited availability of gas-phase metal precursors. The most commonly used reactants for plasma synthesis are solids which have to be evaporated. Another major obstacle is the still poor understanding of the mechanisms by which gas-to-condensed phase transition proceeds in these systems. In this respect, only more recently with the aid of numerical simulation, are the phenomena involving vapour phase nucleation and growth in these high temperature processes being better understood.

This paper presents a review of the research work that has been reported on the thermal plasma synthesis of AlN. It includes the most recent results on the developement of a two-stage transferred-arc reactor, and the results of the investigation of the sinterability of the ultrafine AlN powder produced by this method.

2. The thermal plasma synthesis

A gaseous plasma consists of a mixture of electrons, ions, neutral species in the ground state, excited species, and photons [13]. Overall, negative and positive charges in a plasma balance each other, to produce *quasi-neutrality*. Due to their small mass, electrons have a higher mobility and a natural tendency to have a higher temperature than ions and neutrals (heavy species). Thermal plasma is one type of plasma for which one of the basic conditions to be met is that the temperature of heavy particles approaches the temperature of electrons. The vapour synthesis of AlN using thermal plasmas has been studied by several workers. A summary of the experiments reported in the literature is given in **Table I**. Most of the research

Table I Summary of experimental results reported in the literature for the plasma synthesis of AlN.

Author(s)	Type of Plasma	Power (kW)	Plasma gas		Quenching gas (lpm)	SSA (m^2/g)	Particle size (nm)
			Comp.	Flow rate (lpm)			
Canteloup and Mocellin (1974)	RF	10	Ar	36	NH_3 10.5	220–260	7–8
Baba et al. (1987)	RF	20	Ar/N_2	42/18	NH_3 5~20	50–80	23–37
Wehling et al. (1991)	RF	13	Ar	40	$N_2/NH_3/Ar$ 75	115	30
Etemadi (1991)	DC-TA	2	Ar/N_2 Ar/NH_3	18	Not used	N.A.	30
Godin et al. (1991)	DC-TA	350	Ar/NH_3 NH_3	N.A.	Not used	N.A.	<200
Ageorges et al (1997)	DC-TA	69	N_2/Ar	80/25	NH_3 (100)	12–16	135
Moura and Munz (1997)	DC-TA	8	Ar	15	NH_3/Ar 2/3.6	80–100	23
Da Cruz and Munz (1997)	DC-TA	8.3–12.5	Ar or Ar/H_2	20 18/2	NH_3/Ar (2/2 to 7)	38–270	7–46

RF = Radio frequency induction coupled plasma.
DC-TA = Direct current transferred-arc.
(1) calculated based on reported data.

has been carried out using Al metal solid precursor, either as a powder or as pieces which are melted and evaporated in the plasma and reacted with nitrogen originating from nitrogen gas or ammonia (NH_3). Because of the presence of free charge carriers, in contrast to an ordinary gas, a plasma is electrically conducting. Radio frequency inductively coupled (RF) and transferred-arc plasmas are the two forms of plasma generation that have been most used in vapour phase synthesis applications. A RF plasma, schematically shown in **Figure 1**, operates at frequencies ranging from kHz to MHz. These are electrodeless devices which provide a very clean environment suitable for the production of high purity materials. However, RF plasma generators are usually limited to a few hundred kilowatts of power, and their thermal efficiency rarely exceeds 45%. A free-burning high intensity transferred-arc, schematically shown in **Figure 2**, which most commonly operates on direct current, is one of the simplest forms of thermal plasma generation. Because of their high energy transfer to the anode, transferred-arcs can be efficiently used to melt and evaporate clean metal. Also, transferred-arc systems can be easily scaled up to more than 1 MW with an energy efficiency of more than 85%.

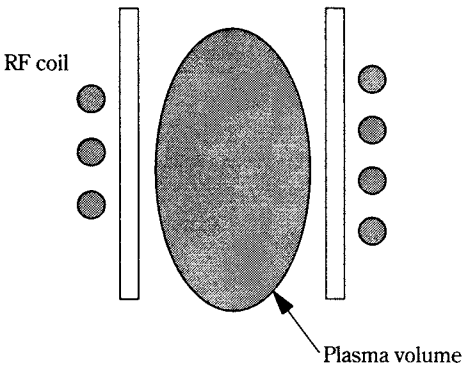

Fig. 1 Radio frequency (RF) plasma.

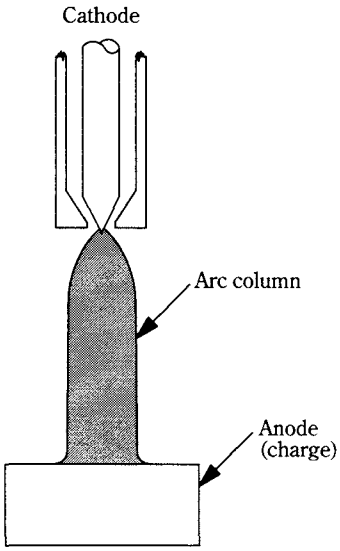

Fig. 2 Transferred-arc plasma.

The use of RF plasma for the preparation of AlN powder from Al powder was reported by Canteloup and Mocellin [14] (1974) and Wehling et al. [15] (1991). A common feature of these two works was the injection of aluminum powder above the fire ball where it evaporated in a non nitriding atmosphere. The nitriding reaction was carried out in a subsequent step, with the injection of NH_3 or NH_3/N_2 mixture in the tail flame. In the reactor used by Canteloup and Mocellin, NH_3 was injected in the tail flame and the reaction products were rapidly quenched. In the reactor used by Wehling et al., NH_3 was also injected in the tail flame, but quenching was not as fast since the reacting mixture flowed in a tube until it was quenched in a large chamber. The faster cooling seems to be the reason why the surface area of the powder produced in the first case was about twice as great. Baba et al. [16] (1987) prepared AlN ultrafine powder using a RF plasma, but used pieces of Al placed on a water cooled copper support which was lifted into the tail flame. Ammonia was injected between the RF coil and the aluminum support.

The use of transferred-arc plasma in nitrogen atmosphere (formed by N_2 or a mixture of N_2 and NH_3) was reported by Etemadi [17] (1991), Godin et al. [18] (1991), and Ageorges et al. [19], [20] (1991, 1993), with the anode formed by an aluminum bath. The AlN produced in these experiments was reported to range from ultrafine particles, collected from the exhaust gases and reactor walls, to large crystal lumps which grew around the anode. Vissokov et al. [21] (1981, 1983) produced ultrafine AlN powder by injecting Al powder near the anode attachment of an electric arc, using a mixture of argon and nitrogen as the plasma forming gas. Cusick (1989) [22] produced AlN using an off-axis transferred-arc reactor with aluminum fed by a consumable anode and N_2 used as plasma gas and nitriding agent. The powder produced by Cusick was reported to be formed of Al and AlN particles. Ageorges et al. [20] reported the use of two different arc devices: (a) transferred-arc on aluminum in N_2 and N_2/NH_3 atmosphere; and (b) cathode and anode arcs coupled together above the aluminum bath. In the second case, NH_3 was inject through a central tube placed above the junction of two arcs.

A great scatter for the particle sizes reported by different authors is observed in the data shown in **Table I**. In the transferred-arc experiments, for which

the metal evaporation and reaction were carried out in the same chamber, longer residence times at high temperatures (as opposed to reaction followed by fast quenching reported for most of the RF systems), and possibly higher concentrations of metal vapour, were available. Both factors might have contributed to producing larger particles than in the RF experiments. Also, because of the nitriding nature of the plasma gas, in these transferred-arc systems, reaction occurred on the surface of the aluminum bath forming undesirable lumps of AlN. Accordingly, the geometry reported by Ageorges et al. for the two arcs coupled above the aluminum bath, allowed for a great deal of recirculation in the plasma chamber. This possibly contributed to the formation of the larger particle sizes reported. Etemadi [17], who used a transferred-arc in a water cooled chamber, and the other authors who used RF equipment, reported smaller particle sizes and more uniform particle size distributions. In general, the use of pure nitrogen gas as the nitriding agent produced low conversion [14], [20]. The examination of the data reported for the above transferred-arc experiments suggests that the control of particle size becomes difficult when the reaction is carried out inside of the plasma chamber. This is due to the steep temperature and concentration gradients which develop around the plasma arc, resulting in different histories for the growth process of individual particles, and to nitridation on the surface of the aluminum bath. Based on these observations, Moura and Munz [23] (1997) proposed a novel concept of transferred-arc reactor for which the reaction was carried out outside the plasma chamber. The powder characteristics reported from their work and the work of Da Cruz and Munz [24] (1997) in a modified version of this reactor concept are also included in **Table I**. This reactor and its reported results are discussed next.

3. Two-stage transferred-arc reactor

The transferred-arc plasma generation system was chosen because it can be easily scaled up to MW power capacity, with a high efficiency [23] – a requirement which has to be met in order to scale up this technology to the many kg/h needed for industrial scale production of these powders. This reactor design provided for the reaction between aluminum vapour and ammonia to be carried out outside the plasma chamber, using NH_3 and Al vapour as reactants. A schematic diagram of the reactor used is shown in **Figure 3**. Al was evaporated from a graphite crucible by a transferred-arc in inert Ar atmosphere. The Al vapour produced in the plasma chamber was transported by the plasma gas into a tubular synthesis reactor. There it reacted with a NH_3/Ar mixture introduced at room temperature through radial jets distributed in the circumference of the tube, close to the entrance of the synthesis reactor. The reaction was further quenched at the end of the reactor by an outward radial flow of room temperature nitrogen. The ultrafine AlN powder produced was collected in a porous stainless steel filter. The production of a 100% converted AlN was reported, with the particles having spherical shape, and specific surface area in the 80 to 100 m^2/g range. These results show that the particle size produced by this reactor configuration was of the same order as that obtained using inductively coupled plasma systems. The mixing conditions at which full conversion could be achieved with this reactor configuration were investigated. It was concluded that the reaction was essentially complete within the mixing zone located a few reactor diameters downstream of the ammonia injection.

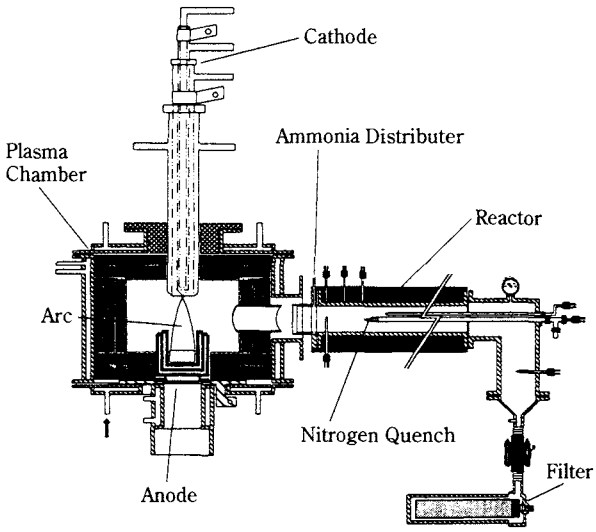

Fig. 3 Schematic diagram of the two-stage transferred arc reactor used by Moura and Munz [23] for the synthesis of AlN ultrafine powder.

The schematic diagram of a more recent design [24] of this reactor concept is shown in **Figure 4**. In this version the transferred-arc chamber and reaction tube were vertically aligned. Modifications were introduced to produce evaporation of Al over relatively long periods of time (from 3 to 5 hr), to promote an homogeneous outflow of the hot gas from the plasma chamber, and to provide space for the introduction of Al vapour concentration and temperature measurement probes. The plasma gas laden with Al(v) flowed through the annular space between the crucible and the plasma chamber wall into the reac-

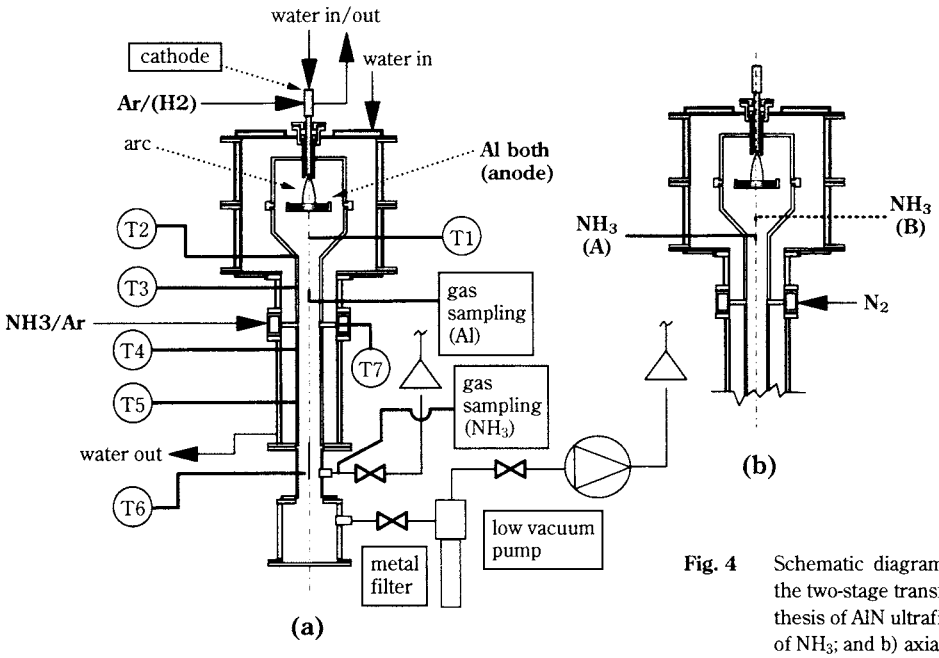

Fig. 4 Schematic diagram of the modified version of the two-stage transferred arc reactor for the synthesis of AlN ultrafine powder: a) radial injection of NH_3; and b) axial injection of NH_3. After [25].

tion tube where it was mixed with NH_3 and quenched by a radial jet of cold gas. This reactor allowed the use of two distinct gas mixing geometries: (a) using radial jets of NH_3/Ar; and (b) the axial injection of NH_3 upstream of the radial quenching point. Typical operating parameters of this reactor are shown in **Table II**. A wide range of particle sizes (38-270 m^2/g) could be produced by modifying the operating parameters such as: plasma gas composition, gas temperature, metal vapour concentration, and nitriding gas mixing geometry.

Table II Typical operating parameters of the transferred-arc plasma and reactive radial gas quenching.

	Radial Injection					Axial Injection	
Plasma gas composition	Ar	Ar		Ar/H_2		Ar	
Plasma gas flow rate (lpm, 298 K)	20	20		18/2		25	25
Temperature T1[1] (K)	1,500	1,800	2,000	1,800	2,000	1,800	2,000
Temperature T3[1] (K)	1,070	1,270	1,410	1,190	1,330	1,300	1,420
Arc length (cm)	5	7	7	5	5	8	8
Arc voltage (V)	29	30	34	50	55	34	36
Arc current (A)	185	275	360	180	250	340	360
Arc Power (kW)	5.4	8.3	12.2	9.0	13.8	11.5	13.0
Quenching:							
Ar (lpm, 298 K)	—	2 to 7				—	
NH_3 (lpm, 298 K)	—	2				—	
N_2 (lpm, 298 K)		—				6	
Pressure	100 kPa						

[1] Thermocouples were positioned according to Figure 4.

The specific surface area of the powders produced under different conditions with the radial and axial injection of NH_3 is shown in **Figures 5** and **6**, respectively. The finest powders were produced with the radial injection of ammonia, using Ar plasma gas with a temperature of 1800 K, and low quenching intensity. The coarsest powders were produced with a plasma chamber off-gas temperature of 2000 K, and the axial injection of pure (not mixed with Ar) NH_3 at a position that provided the longest residence time of particles at the high temperature region.

A theoretical analysis of these operating conditions was carried out using a numerical model [25]. The model, which accounted for the nucleation of particles from the gas phase and growth by condensation, reaction, and coagulation, showed that the nucleation rate in the AlN system is largely dominated by heterogeneous reaction, possibly on the surface of subcritical particles. The many experimental trends were studied in light of the modelling analysis. The opposing trends observed with the radial injection of NH_3 (**Figure 5**) were shown to be associated with the nucleation of particles under different concentrations of nitriding agent. Typical modelling results for the nucleation rate using the radial injection of NH_3 are shown in **Figure 7**. Except for the plasma gas composition, the operating conditions used in the simulation were: gas temperature of 2000 K at the entrance of the tubular reactor and a radial jet formed by 2 lpm of NH_3 and 5 lpm of Ar. Temperature gradients caused by heat loss upstream the point where NH_3 is injected result in the nucleation of pure Al particles near the

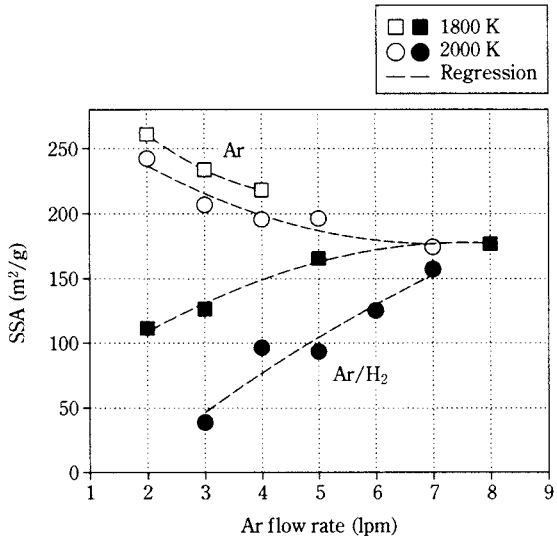

Fig. 5 Specific surface area of powders produced with the radial injection of NH_3, as a function of the impinging jet intensity, and plasma gas temperature and composition. NH_3 flow rate is kept constant at 2 lpm, and the jet intensity is modified by changing the amount of also injected Ar.

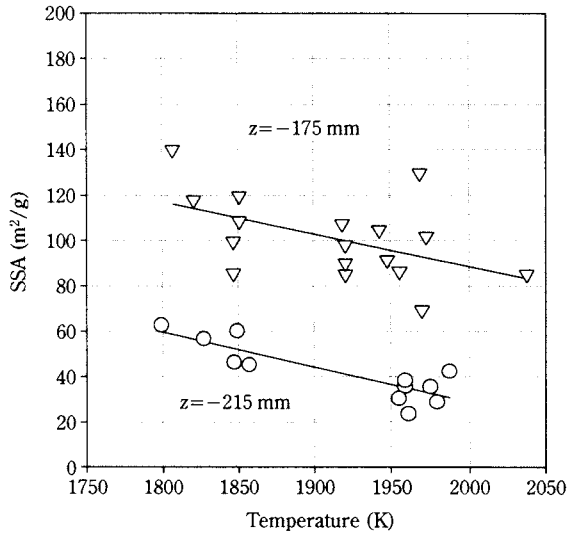

Fig. 6 Specific surface area of powders produced with the axial injection of NH_3, as a function of the gas temperature at the entrance of the tubular reactor, and position of NH_3 injection upstream of the quenching jet (z=0 mm).

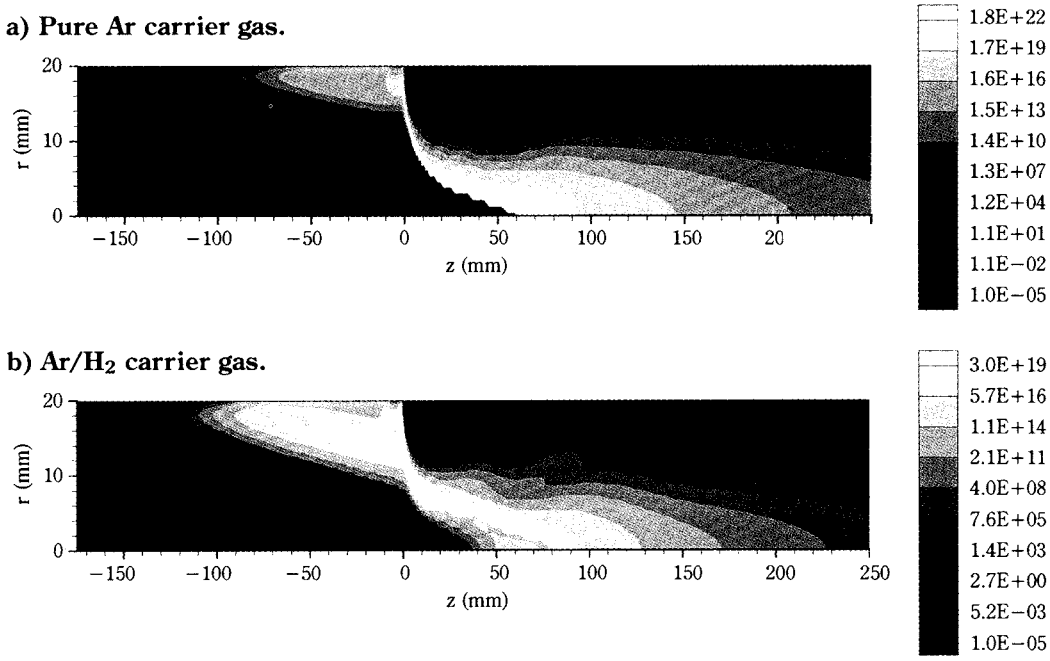

Fig. 7 Nucleation rate ($1/m^3/s$) predicted for different plasma gas compositions.

tube wall, in the absence of nitriding agent. A higher heat loss produced by the greater thermal conductivity of the Ar/H_2 gas mixture (in contrast with pure Ar) causes nucleation in a broader volume in this region [**Figure 7 (b)**]. The modelling analysis also showed that the change of particle size with temperature resulted from the greater Al(v) concentration produced with higher temperatures of the plasma chamber off-gas.

The modelling predicted weight percentage of AlN in the forming particles along the reaction zone for the radial and axial injection of ammonia of pure Ar plasma gas at 2000 K are shown in **Figure 8 (a)** and **(b)**, respectively. Particles which nucleate near the wall upstream of the jet do so in total absence of NH_3. Those particles which pass the injection point and remain near the wall where the NH_3 concentration is high, but temperature and Al concentration are rather

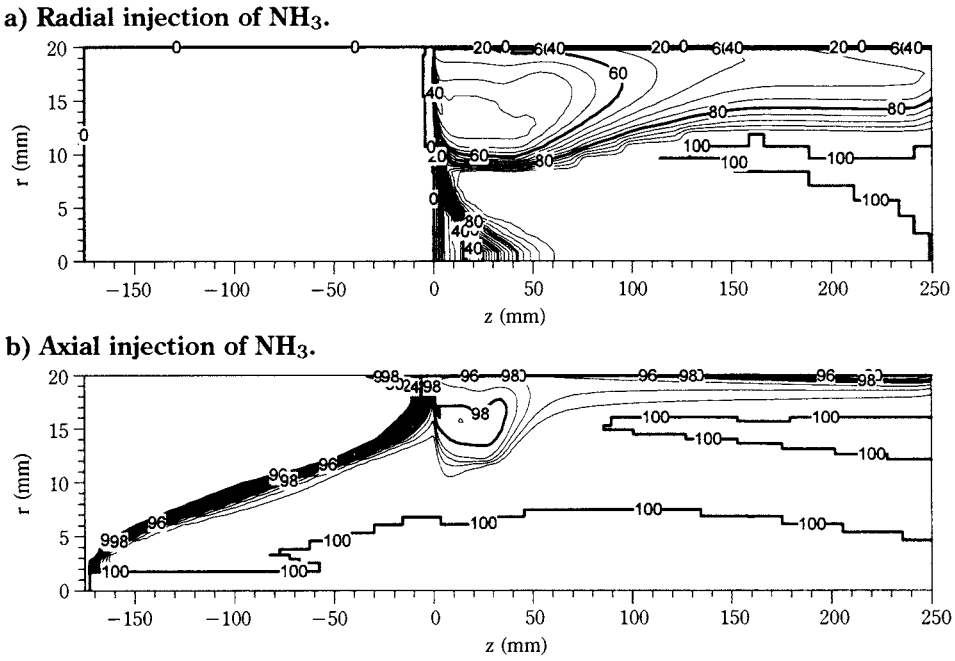

Fig. 8 AlN content in the powder predicted by the numerical simulation (Ar plasma, 2000 K): a) with the radial injection of NH_3 (2 lpm); and b) with the axial injection of NH_3 at $z=-175$ mm, and radial injection of N_2 (6 lpm) at $z=0$ mm.

low, have their already condensed Al only partially converted to AlN. Those particles which form in the higher temperature central portion, will become fully nitrided far before the end of the exit of the calculation domain. On the other hand, in the axial injection model [**Figure 8 (b)**], it is observed that full conversion is readily achieved in the high temperature and high NH_3 concentration core. As in the the radial injection, because of the lower temperatures near the tube wall, the particles which form in this region are difficult to nitride fully. However, in contrast with the radial injection, the conversion at this region is still better than 96%. Since most of the gas-to-condensed phase transition occurs in the hot core, a conversion of 99.9% is predicted for the average of the powder at the exit of the calculation domain in the 2000 K temperature level. The modelling results of the 1800 K temperature level showed similar distributions. These results, which are in full agreement with the experiments, confirm the importance of avoiding excessive cooling rates in the mixing zone in order to obtain fully converted powders.

3.2. Powder characterization

The examination of the AlN ultrafine powders produced by plasma using Scanning Electrons Microscopy (SEM) [15], [23] and Atomic Force Microscopy (AFM) [23] showed loose agglomerates of rounded particles. TEM micrographs of AlN powders synthesized by plasma are shown in **Figure 9** [25]. The larger sizes and broader size distribution observed in **Figure 9 (b)** are typical of longer residense times in a hot reaction zone. In contrast, the TEM micrograph

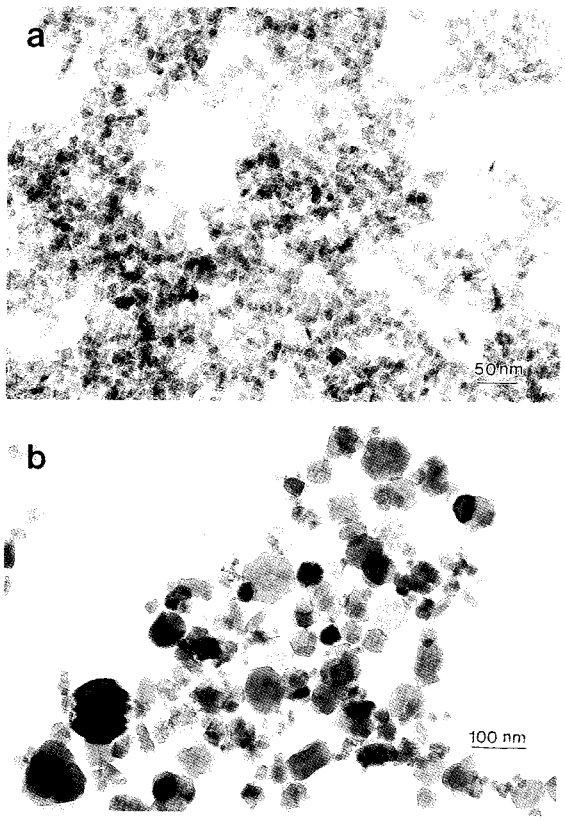

Fig. 9 TEM micrographs of powder produced with: a) the axial injection of NH_3 in the central portion of a tubular reactor; and b) the radial injection of NH_3 mixed with Ar. After [25].

of powders which where produced with a more stringent cooling condition, and therefore shorter residence times, shows smaller particles of a narrower size distribution [**Figure 9 (a)**].

Oxygen is the major impurity commonly found in these ultrafine powders; most authors have attributed this to contamination during handling in atmospheric air, after the powder has been produced. The oxygen content of nanosize AlN powders, plotted as a function of their specific surface area, is shown in **Figure 10**. For illustration, the data reported by Hashimoto and Yoden [26] (1992), who synthesized AlN ultrafine powders from aluminum chloride and glucose, is also included in this figure. The positive correlation between oxygen and surface area is evident. The results of Fourier Transform Infrared Spectroscopy (FTIR) [16], [23], [25] showed broad peaks of the O-H bond on the surface of the powder, suggesting that the high specific surface area of AlN might make it very reactive with moisture in the air. The presence of carbon, reported for the powders produced with transferred-arc systems, was attributed to the erosion of the graphite crucible by the plasma arc. The x-ray photoelectron spectroscopy (XPS) analysis of the AlN powders produced using the two-stage transferred-arc reactor also showed the presence of carbon contamination resulting from the graphite crucible erosion [25].

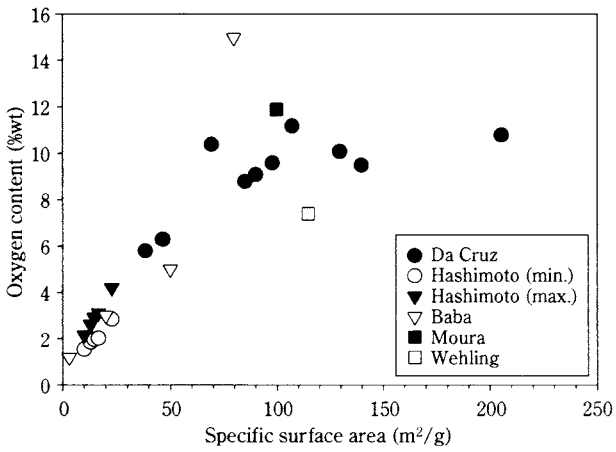

Fig. 10 Oxygen content of nanosize AlN powders, plotted as a function of their specific surface area.

3.3. Powder sinterability

Commercially available powders produced by the carbothermal reduction of Al_2O_3 have been reported to sinter at temperatures above 1850°C, using hot pressing sintering [12]. The preparation of full density AlN ceramics by pressureless sintering of AlN ultrafine powders produced by plasma, with and without sintering aids, has been reported [15], [16], [20], [27]. Presureless sintering without additives at temperatures as low as 1530°C has been reported [15].

The densification and microstructure development of AlN powders with a specific surface area in the 85-108 m^2/g range, produced in the two-stage transferred-arc plasma reactor, were reported by Da Cruz et al. [29]. Powder samples were compacted as produced, and pressureless sintered in the 1250°C to 1750°C range, for 3 hours. The fractured surface of a sample sintered at 1350°C, examined by TEM using the Pt/C replica and shadowing technique, is shown in **Figure 11**. This represents an intermediate stage of the sintering process in which fine grains of the starting powder and a plate-like structure, observed to develop at scattered domains, are present. Sintering at 1750°C resulted in the fully densified structure shown in **Figure 12**. The XRD patterns of the starting powder and of samples which have been sintered at 1750°C are shown in **Figure 13**. Peaks of a second

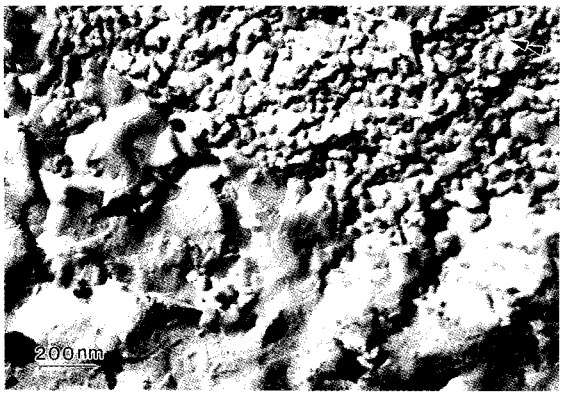

Fig. 11 TEM image of Pt/C replica of the surface of a fragment of AlN ultrafine powder sintered at 1350°C. A plate like structure is observed to develop. The arrow indicates the shadowing direction.

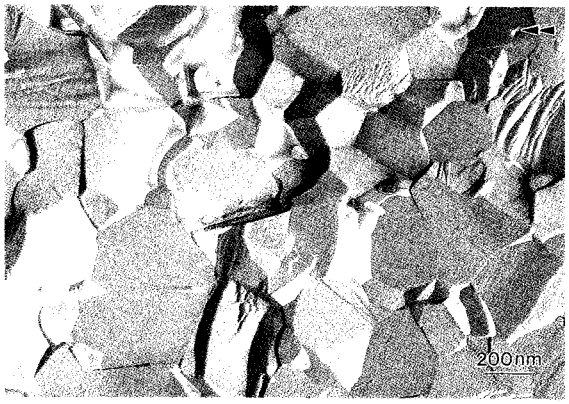

Fig. 12 TEM image of Pt/C replica of the surface of a fragment of AlN ultrafine powder sintered at 1750°C. The arrow indicates the shadowing direction.

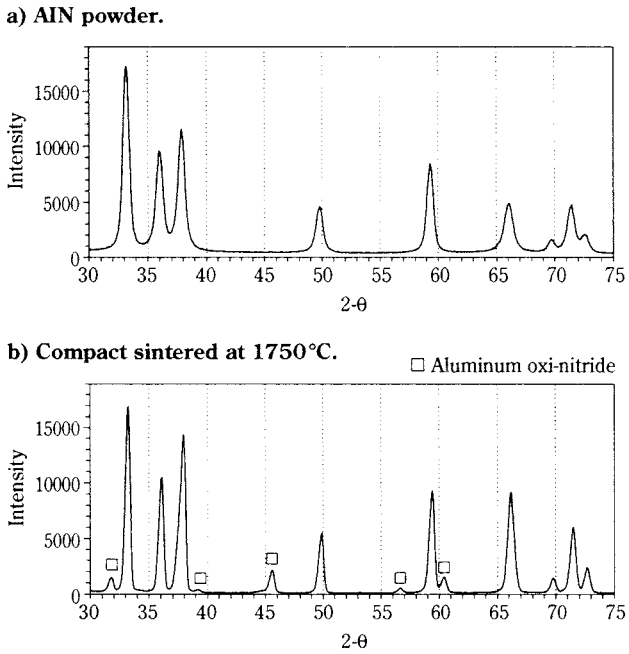

Fig. 13 X-ray diffraction pattern: a) AlN powder; b) sample sintered at 1750°C.

phase, identified as $Al_{8/3-x/3}O_{4-x}N_x$ (JCPD card 18-52), observed in the pattern of the sintered sample [**Figure 13 (b)**], possibly resulted from the incorporation of the oxygen from the surface of the starting particles into the sintered material structure during densification. Because the pseudo-binary solution AlN-Al_2O_3 presents high melting points at any composition [28], and oxygen content has been observed to exert little influence on AlN densification [26], it is believed that the densification of these fine powders occurs by solid state sintering, driven by the large surface free-energy change upon sintering rather than by liquid phase formation associated with their relatively high oxygen content.

4. Conclusions

An overview of the thermal plasma synthesis of ultrafine AlN powders has been presented. Laboratory scale experiments, using inductively coupled and transferred-arc plasma systems, have been used to produce very fine powders. Sinterability experiments have demonstrated that these powders can be sintered at lower temperatures than powders produced by conventional methods. The FTIR exam of the powder surface suggests that the high specific surface area of AlN may make it very reactive with moisture in the air. Therefore, to produce commercially acceptable powders aimed to high conductivity application, surface oxidation must be prevented by avoiding contact of the powder with atmosphere before final parts are produced. The presence of carbon observed in powders prepared with transferred-arc systems remains a problem to be solved, possibly with improved design of molten metal holders.

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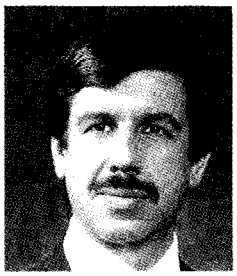

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