

Processing of in-situ SiC reinforced AlN based composites

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In this study, highly dense (>97%) in-situ SiC reinforced AlN based ceramic-metal composites were produced by pressureless reactive infiltration of 7075 aluminium alloys into porous α -Si₃N₄, α -Si₃N₄ + 4 wt. % carbon and α -Si₃N₄ + 8 wt. % carbon preforms at 1400 °C for 4 hours under an argon gas atmosphere. X-ray diffraction and transmission electron microscopy analysis of the designed α -Si₃N₄-Al and α -Si₃N₄-C-Al composites revealed that Si₃N₄ was completely consumed during the infiltration process via reacting with the Al alloy and resulted in the formation of AlN and Si phases. The aim of the current work was to introduce a novel method to produce in-situ SiC reinforced AlN based ceramic-metal composites. Composites with relatively low metal contents were successfully fabricated through incorporating active carbon to the starting α -Si₃N₄ powder, which in turn consumes the liberated metallic Si phase in the system via chemical reactions leading to the formation of SiC ceramic phase. Formation of in-situ SiC, AlN and metallic Si were also confirmed by analytical transmission electron microscopy investigations.

Key words: Ceramic-Metal Composites, Si₃N₄, AlN, SiC, Reactive Infiltration, In-Situ Reactions.

Introduction

Properties of AlN

AlN has a high thermal conductivity (ranging from 30-260 W/m.K for polycrystalline materials to 320 W/m.K for single crystals) [1], a low coefficient of thermal expansion ($4.5 \times 10^{-6}/^{\circ}\text{C}$) which closely matches that of silicon ($4.7 \times 10^{-6}/\text{K}$) [2], a high electrical resistivity (10^{13} W.cm) [2], good mechanical strength, a large band gap (5.4-6.3 eV) [3], excellent corrosion resistance to plasma and halogen gases [2], a relatively low dielectric constant (8.7 at 1 MHz) [4], a relatively high hardness (1300 kg/mm²) [3], a high melting temperature (2000 °C) [3], sufficiently high temperature compatibility with refractory metals, good resistance to thermal shock and good oxidation resistance at elevated temperatures [1, 2, 4-9]. AlN is also an ecologically safe material [10].

Limitations of AlN

The widespread use of AlN, however, has been limited for several reasons. First, the production of AlN based ceramics by traditional means (e.g., sintering) requires sub-micrometer AlN powders that are presently expensive which is a great obstacle to its widespread usage. [11] Also high-cost capital equipment is required to sinter AlN in a controlled atmosphere at temperatures of

around 1800 °C [12]. Second, AlN powder hydrolyzes easily when exposed to an aqueous environment or humidity forming aluminium hydroxide. Thus, all powder processing must be done in a non-aqueous environment and with extreme care. Another drawback is its reputation for poor lot-to-lot reproducibility, in terms of thermal conductivity and substrate metallization problems [1]. These factors, among others, have delayed the expected market growth for the material [2]. Thus, there is a strong incentive for the development of more-efficient production processes [2].

Properties of Al

It is well known that ceramics are very brittle materials and the low toughness of these materials can be improved by in-situ production of elongated grains [13, 14] or by the addition of a ductile phase such as metals and especially aluminium and its alloys [15-26]. As a result, ductile metal phase reinforced high-volume fraction (> 50%) ceramic matrix composites are emerging as an important class of materials with potential for structural applications where high specific modulus, strength, and superior wear characteristics are desirable [27, 28].

Aluminium and its alloys are one of the most popular metals for the fabrication of AlN based ceramic-metal composites due to their high thermal conductivity (180-230 Wm⁻¹K⁻¹), low density (2.7 gcm⁻³), capability to be strengthened by precipitation hardening, good corrosion resistance, electrical conductivity, and high damping capacity. [24, 29-32] Aluminium alloys are also easier to process, compared with other metals having a high

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thermal conductivity such as copper [29]. The major drawback of pure aluminium is its high coefficient of thermal expansion ($\alpha = 23.10^{-6} \text{K}^{-1}$), but it can be decreased to about 18.10^{-6}K^{-1} via addition of Si. AlN reinforced Al alloy composites possess a high elastic modulus [33]. Moreover, coefficient of thermal expansion for this system is similar to silicon and it can be used in semiconductor packaging in aerospace structures. Imai et al. [34] showed that this composite exhibits high strain rate superplasticity.

AlN based ceramic-metal composites

AlN-Al composites combine the attractive properties of AlN with the excellent ductility, conductivity and low density of Al [35]. Thus, these composites are good candidates for either structural or electronic applications [30, 36, 37]. Several fabrication methods have been reported for the fabrication of AlN based ceramic-metal composites [38]. Among all these techniques pressureless melt infiltration method has various advantages such as providing a uniform distribution of the reinforcement, net- or near-net-shaping. It is relatively easy to control the volume fraction of reinforcements and ceramic-metal composites with more than 50 vol. % ceramic reinforcements can be fabricated to meet special properties, i.e., higher stiffness and wear resistance, or a coefficient of thermal expansion close to that of steel components [39].

Aghajanian et al. reported a novel two-step infiltration and reaction approach to produce AlN-Si-Al composites with or without SiC reinforcements [40]. All preforms, containing either 100% Si_3N_4 or 70% Si_3N_4 + 30% SiC, were prepared by conventional uniaxial pressing. XRD results showed that Al reacted with Si_3N_4 to form AlN and Si. The advantage of this fabrication process is that it uses an economical filler material and a relatively low processing temperature. In the production of AlN based ceramic-metal composites via reacting Si_3N_4 with Al by pressureless infiltration technique, a significant amount of the metal source is consumed and accompanied by the release of metallic Si at appreciable quantities. Metallic Si has a higher hardness when compared with Al. However, it is a very brittle material.

Motivation of the study

The motivation for conducting the present work was to investigate the possibility of producing an in-situ SiC reinforced AlN based composite with reduced residual metal content. In order to achieve this goal, active carbon was added to the system to enable consumption of the released Si, a by-product of the reaction between Si_3N_4 and Al, via formation of SiC. Furthermore, the effect of active C addition on the phase formation and microstructure development of AlN based ceramic-metal composites was investigated with X-ray diffraction and advanced transmission electron microscopy techniques.

Experimental Procedure

Silzot HQ $\alpha\text{-Si}_3\text{N}_4$ ($d_{50} \approx 2 \mu\text{m}$) and active carbon ($\leq 60 \text{ nm}$) starting powders were mixed by wet milling in a planetary ball mill in isopropanol alcohol medium and then dried in a rotary evaporator to achieve designed batches. Designed sample compositions are given in Table 1.

Porous ceramic preforms were prepared by uniaxially pressing the powder mixtures at 175 MPa. Ceramic-metal composites were produced by melt infiltrating 7075 Al alloy blocks (Table 2) into porous ceramic pellets under an Ar gas atmosphere at 1400 °C for 4 hours. Heating rate was 5 °C/min up to 900 °C and 10 °C/min up to 1400 °C. Cooling rate from the infiltration temperature to 900 °C was 10 °C/min and then 5 °C/min down to the room temperature.

Bulk density values of specimens were calculated by Archimedes' displacement method. Phase analysis of the fabricated composites was performed by using an X-ray diffractometer (Rigaku Rint 2200, Tokyo, Japan) with monochromatic $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) at a scan speed of 0.5 °/min. Microstructure investigations were carried out by using a Schottky emitter field emission gun (FEG) SEM (Zeiss SUPRA 50 VP). Samples were prepared through a conventional procedure, using mechanical polishing steps, followed by Ar-ion beam thinning (Baltec™ RES 101) for TEM investigations. Afterwards, the samples were characterized by using a 200 kV field emission TEM (Jeol™ JEM-2100F) equipped with scanning transmission electron microscope high angle annular dark field (STEM-HAADF) detector (Model 3000, Fischione), electron energy loss spectrometer (EELS) and energy filter (Gatan™ GIF Tridiem).

Thermodynamic data of the relevant reactions and phase diagrams of the system components were taken

Table 1. Designed sample compositions.

Composition	Si_3N_4 (wt. %)	C (wt. %)
100SN	100	–
96SN4C	96	4
92SN8C	92	8

SN: Si_3N_4 , C: active carbon.

Table 2. Chemical composition, melting and boiling point of the alloy constituents.

Element	Al	Zn	Mg	Cu	Fe	Cr
wt. %	90.4	5.3	2.3	1.3	0.4	0.3
Melting temperature, °C	660	420	649	1083	1535	1857
Boiling temperature, °C	2467	907	1107	2567	2750	2672

from the MTDATA thermodynamic program.

Results and Discussion

Green and bulk density

Green densities of the porous ceramic preforms are given in Table 3. When 4 wt. % active C was added to the α - Si_3N_4 powder, a slight increase in the green density, was observed. However, when the used active C amount was increased to 8 wt. % no improvement was observed in the green density probably due to the increased amount of finer starting powder content. Bulk density and residual open porosity contents of the fabricated ceramic-metal composites are also given in Table 3. Average bulk density of the active C containing samples were slightly lower than that of the 100SN sample due to the somewhat higher amount of open porosity content in the former ones.

Phase analysis

XRD patterns of the specimens are represented in Fig. 1. As clearly seen from the XRD results there is no metallic Zn and Mg phase detected, despite the fact that 7075 Al alloy contains these alloying elements in appreciable amounts. This may be explained as follows: Since the boiling point of both Zn and Mg are lower (Table 2) than the infiltration temperature (1400 °C) selected in this study, it is very likely that both have been volatilized during the infiltration process.

XRD results of the 100 SN sample revealed that it consists of AlN, Al, Si and CuAl_2 phases. AlN and metallic Si phases are formed as a result of the reaction between Si_3N_4 and Al during the infiltration process. Also it is determined from the XRD results that the starting α - Si_3N_4 powder was practically completely consumed during the infiltration process via reacting with the Al alloy (Eq. 1). Al-N and Si-N phase diagrams shown in Fig. 2 indicate that N has no solubility, in either Al or Si, indicating that the released N coming from the dissolution of Si_3N_4 must immediately react with Al, possibly forming the AlN phase via the exothermic reaction (Eq. 1) given below.

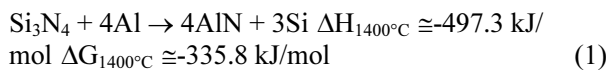


Table 3. Green density of the ceramic pellets, bulk density and open porosity content of the fabricated ceramic-metal composites.

Composition	Green density* (%)	Bulk density* (g/cm^3)	Open porosity* (%)
100SN	44.3 ± 0.4	2.89 ± 0.05	0.9 ± 0.2
96SN4C	46.9 ± 0.1	2.82 ± 0.01	1.9 ± 1.0
92SN8C	44.1 ± 0.2	2.82 ± 0.02	2.3 ± 0.3

*: average of 3 sample measurements.

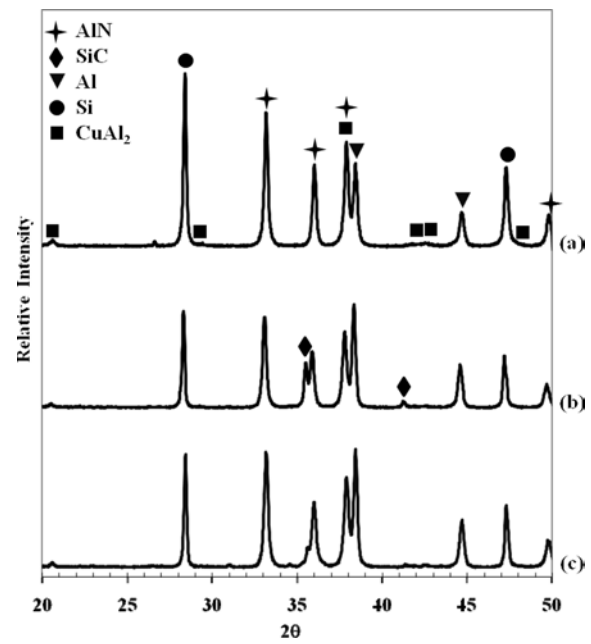


Fig. 1. XRD patterns of (a) 100SN, (b) 92SN8C and (c) 96SN4C ceramic-metal composites.

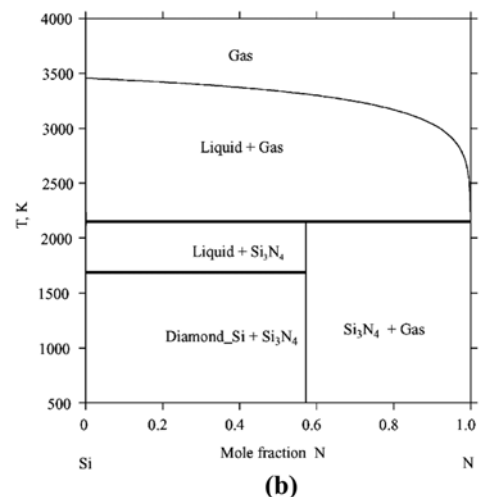
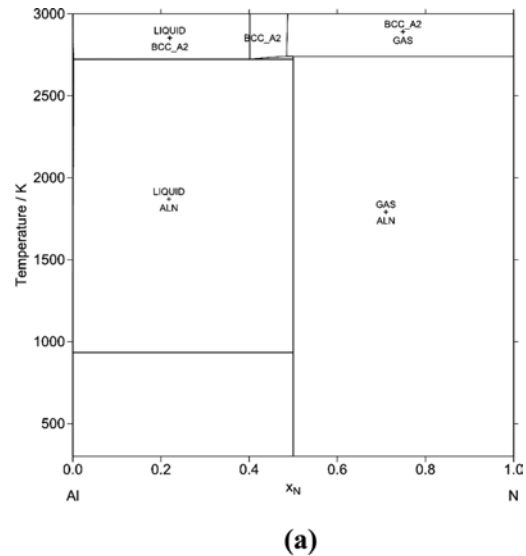


Fig. 2. (a) Al-N and (b) Si-N phase diagrams.

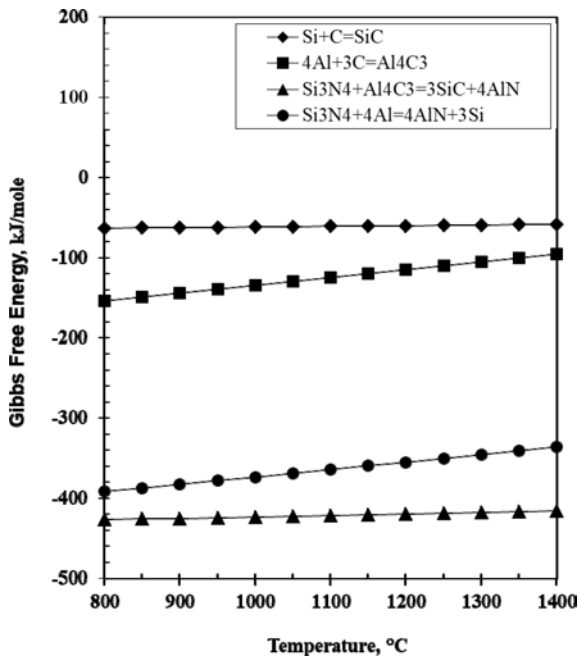
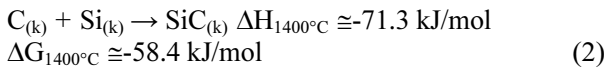


Fig. 3. Comparative plot of Gibbs free energy versus temperature graphs for the suggested reactions resulting in the formation of SiC, Al₄C₃, AlN and Si.

Analysis of the residual Al alloy block that remained on the surface of the infiltrated preform, revealed the existence of needle like Si crystals, ranging from several hundred μm to mm in size. This phenomenon may be attributed to the diffusion of metallic Si liberated from the reaction between $\alpha\text{-Si}_3\text{N}_4$ and Al through the interconnected liquid metal channels during the infiltration process. Further growth of the Si crystals may have occurred during the cooling step.

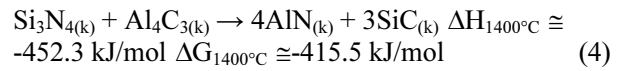
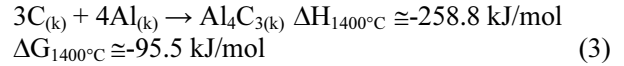
Phase analysis of the active C containing samples indicated that besides the phases of AlN, Al, Si and CuAl₂, in-situ SiC was formed during the infiltration process. Two routes may be proposed to explain the formation of in-situ SiC.

i) SiC formation may have occurred as a result of the reaction between liberated metallic Si and nano-size active C. Indeed, thermodynamic data (Fig. 3) confirm that there is a driving force for the free active C to react with the released metallic Si (Eq. 2).



ii) Al has a high affinity for C. From a thermodynamic point of view, Al₄C₃ may have been produced through Eq. 3. Al₄C₃ formation seems to be more likely compared to the formation of SiC via reaction 2, since the former one has a more negative ΔG value. However, there is a strong driving force for Si₃N₄ to react with Al₄C₃ phase to produce both AlN and SiC phases, too (Eq. 4). This may also be the

reason why the existence of Al₄C₃ could not be verified by XRD analyses. The reactions given in Eq. 2 and 3 are both exothermic like Eq. 4. But compared with Eq. 4 their driving force is much lower (Fig. 3). Presence of Al₄C₃ could not be verified by XRD measurements of the active C containing samples. Yet, it is reasonable, to propose that although Al₄C₃ forms during the infiltration process, it may be consumed via reacting with the Si₃N₄ phase due to the relatively high exothermic nature of this reaction (Eq. 4).



Phase quantification

Amount of metal and ceramic phase content of the specimens were calculated according to Eq. 1 and 2 by making use of the green density values of the porous $\alpha\text{-Si}_3\text{N}_4/\alpha\text{-Si}_3\text{N}_4 + \text{C}$ pellets. Phase content of the samples after infiltration was calculated based on the following assumptions.

- Voids of the porous $\alpha\text{-Si}_3\text{N}_4$ / $\alpha\text{-Si}_3\text{N}_4 + \text{C}$ preforms are completely filled with 7075 Al alloy during the infiltration step.
- $\alpha\text{-Si}_3\text{N}_4$ and active C starting powders are consumed completely (Fig. 1) during the infiltration process according to Eq. 1 and 2, respectively.
- Diffusion of the liberated Si to the excess Al block remaining on the sample surface is negligible.
- Phase assemblage of the 100SN sample includes AlN, Al and Si, whereas that of the active C containing samples includes AlN, SiC, Al and Si (Fig. 1).
- The amount of CuAl₂ forming in the composition is negligible.

Phase content of the samples were calculated at the

Table 4. Calculated phase content of the fabricated ceramic-metal composites at given green density values.

	100SN	96SN4C	92SN8C
Green Density (%)	44.3	46.9	44.1
Al (vol. %)	14.5	12.3	20.3
Si (vol. %)	35.8	30.6	21.9
Total metal content (vol. %)	50.2	43.0	42.2
Decrease in total metal content (%)		14	16
AlN (vol. %)	49.8	50.9	46.1
SiC (vol. %)	—	6.2	11.7
Total ceramic content (vol. %)	49.8	57.01	57.8
Increase in total ceramic content (%)		14	16

Table 5. Relative peak intensity ratio of the present phases in the fabricated composites.

Phases	Peak Intensity (cps)			FWHM		
	100SN	96SN4C	92SN8C	100SN	96SN4C	92SN8C
Al ^θ	983	1409	1270	0.321	0.310	0.297
AlN ^σ	3982	3704	2694	0.261	0.284	0.273
Si*	5133	4028	2863	0.203	0.181	0.190
SiC ^Δ	0	973	1303	0.000	0.281	0.313

Phases	Areal Peak Intensities		
	100SN	96SN4C	92SN8C
Al ^θ	316	436	378
AlN ^σ	1040	1052	736
Si*	1042	730	544
SiC ^Δ	0	274	408

Intensity ratio of selected phases							
	100SN	96SN4C	92SN8C		100SN	96SN4C	92SN8C
Si*/Al ^θ	3.3	1.7	1.4	Al ^θ /SiC ^Δ	—	1.6	0.9
Si*/SiC ^Δ	—	2.7	1.3	Al ^θ /AlN ^σ	0.3	0.4	0.5
Si*/AlN ^σ	1.0	0.7	0.7	AlN ^σ /SiC ^Δ	—	3.8	1.8

PDF card numbers of the phases and 2θ values of the selected peaks are as follows:

*: 28.443 (Si, 00-027-1402), ^θ: 38.473 (Al, 00-004-0787), ^σ: 33.216 (AlN, 00-025-1133) and ^Δ: 35.597 (SiC, 00-029-1129).

mentioned conditions and achieved results are given in Table 4. The following conclusions can be drawn according to these calculations:

- It is observed that the total ceramic content of the samples increased considerably via in-situ SiC phase formation, by 14 and 16% for 96SN4C and 92SN8C samples, respectively.

- Si phase content decreased significantly in the active C containing samples, by 14 and 39% for 96SN4C and 92SN8C samples, respectively.

- With the use 8 wt. % active C, residual Al content in the composite increased considerably compared to 100SN and 96SN4C samples possibly due to the decreased amount of initial α -Si₃N₄ powder content. The reaction between Si₃N₄ and Al results in a substantial volume expansion of ~4%. Furthermore, this reaction assists the reduction of the metal phase in the final composite. When 1 mole Si₃N₄ reacts with 4 moles of Al (Eq. 1), eventually total ceramic phase consequently decreases from 47.9 vol. % to 41.8 vol. %.

Amount of the total ceramic is insensitive to the amount of active carbon added in the studied range. However, the amount of the in-situ formed SiC content of 92SN8C is almost twice that of 96SN4C (Table 4). When the amount of the in-situ formed SiC phase is increased in the sample while preserving total ceramic phase content, composites are expected to exhibit more isotropic properties due to the achievement of a more homogenous SiC distribution in the microstructure.

Relative areal peak intensity ratios of the current phases deduced from the XRD data depicted in Fig. 3 are given in Table 5. The degree of agreement of these

data with the calculated phase contents of the samples given in (Table 4) are observed to depend on the green density. Conclusions that are deduced from the peak areal intensity ratio of the current phases are as follows;

- Si/Al ratio is decreased sharply from 3.3 (100SN) to 1.7 and 1.4 for 96SN4C and 92SN8C samples, respectively via in-situ SiC formation.

- When the amount of active C is increased from 4 wt.% to 8 wt.% Al/SiC, AlN/SiC and Si/SiC peak intensity ratio is decreased by ≥ 50%. Calculated amount of in-situ formed SiC is significantly higher in 92SN8C specimen (Table 4). Thus, these decreased peak intensity ratios are as expected.

Microstructure development

SEM fracture surface investigations of all the samples revealed similar features; a dense and very fine (< 1 μm) microstructure with a brittle fracture mode of fine AlN particles, and a ductile failure mode of the metallic Al (Fig. 4). TEM techniques were used to investigate the fine grained microstructure of the samples.

STEM image in Fig. 5 shows the general microstructure of the 92SN8C composite. It is observed that the composite has a very fine microstructure in the sub-micron range. Atomic number contrast HAADF image shows the different phases in the sample, white regions contain metallic Si while the other regions are either SiC or AlN grains.

Fig. 6 shows EFTEM 3 window elemental mapping images of the 92SN8C composite.

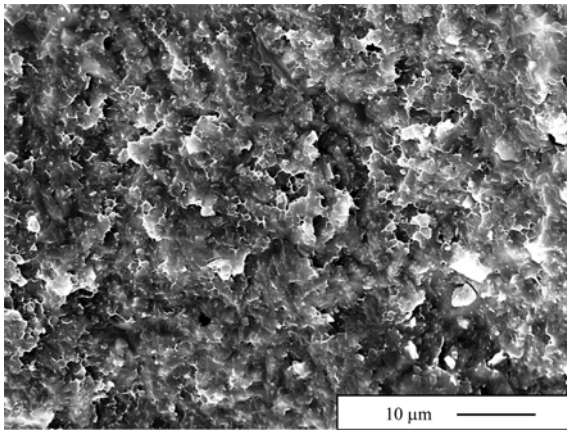


Fig. 4. Fracture surface of 92SN8C composite.

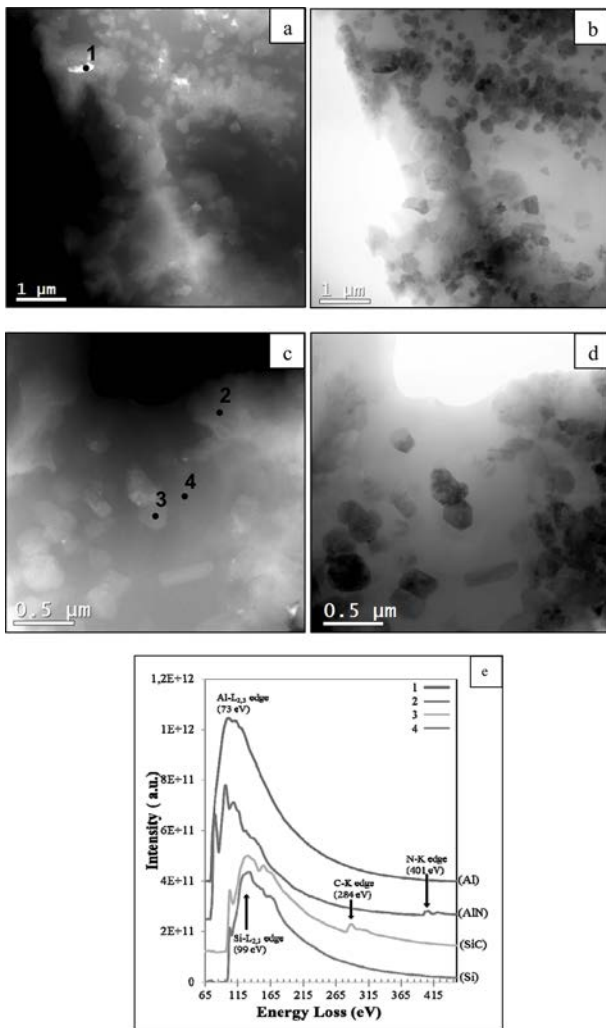


Fig. 5. STEM images taken from the sample: (a) is a HAADF image and (b) is the corresponding BF image of (a). (c) and (d) are higher magnification images. (e) is the EEL spectrum collected from the regions labelled as “1”, “2”, “3” and “4”.

Conclusions

- Highly dense (> 97%) and fine grained (~ 200-300 nm) AlN based ceramic-metal composites were

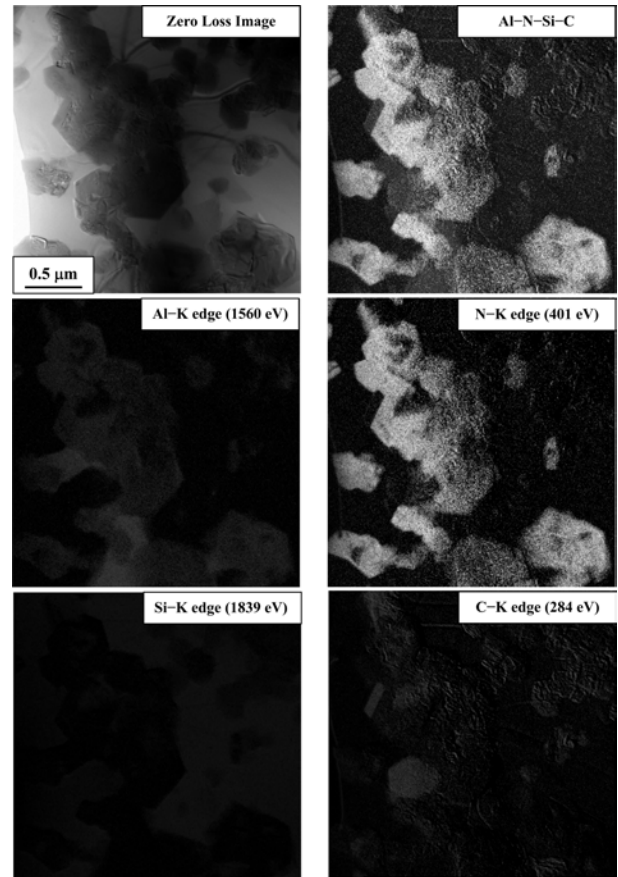


Fig. 6. EFTEM 3 window elemental mapping showing Zero loss image (± 20 eV) and general distribution of the elements in the 92SN8C composite.

produced by pressureless reactive infiltration of 7075 aluminium alloy into porous α - Si_3N_4 (100SN) and α - Si_3N_4 + carbon (96SN4C and 92SN8C) pellets at 1400 °C for 4 hours under an argon gas atmosphere.

- Depletion of Al by reacting with α - Si_3N_4 during the infiltration process resulted in the formation of a cost effective non-oxide ceramic-metal composite containing AlN and free metallic Si as the reaction products. Incorporating active C into the starting α - Si_3N_4 powder, results in the consumption of the liberated metallic Si phase in the system via reactions leading to the formation of in-situ SiC ceramic phase. Produced composites have a relatively high-volume fraction of ceramic phase (~57%) that may find potential use for structural applications where high specific modulus, strength, and wear characteristics are desirable.

- Si phase content decreased significantly in the active C containing samples, by ~14 and ~39% for 96SN4C and 92SN8C samples, respectively. Decrease in total metal content (Si + Al) for 96SN4C and 92SN8C samples were calculated to be ~14 vol. % and ~16 vol. %, respectively.

- Based on phase analysis results of the produced composites, the data obtained from literature and

MTDATA thermodynamic package program, the sequence and likelihood of possible reactions was evaluated. These considerations led to the conclusion that SiC, besides a reaction between Si and C, may have also formed by the reaction between formed Al_4C_3 and starting Si_3N_4 powder.

Acknowledgments

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