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Template-Based Synthesis of Aluminum Nitride Hollow Nanofibers Via Plasma-Enhanced Atomic Layer Deposition

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Aluminum nitride (AlN) hollow nanofibers were synthesized via plasma-enhanced atomic layer deposition using sacrificial electrospun polymeric nanofiber templates having different average fiber diameters (~70, ~330, and ~740 nm). Depositions were carried out at 200°C using trimethylaluminum and ammonia precursors. AlN-coated nanofibers were calcined subsequently at 500°C for 2 h to remove the sacrificial polymeric nanofiber template. SEM studies have shown that there is a critical wall thickness value depending on the template's average fiber diameter for AlN hollow nanofibers to preserve their shapes after the template has been removed by calcination. Best morphologies were observed for AlN hollow nanofibers prepared by depositing 800 cycles (corresponding to ~69 nm) on nanofiber templates having ~330 nm average fiber diameter. TEM images indicated uniform wall thicknesses of ~65 nm along the fiber axes for samples prepared using templates having ~70 and ~330 nm average fiber diameters. Synthesized AlN hollow nanofibers were polycrystalline with a hexagonal crystal structure as determined by high-resolution TEM and selected area electron diffraction. Chemical compositions of coated and calcined samples were studied using X-ray photoelectron spectroscopy (XPS). High-resolution XPS spectra confirmed the presence of AlN.

I. Introduction

III-NITRIDE compound semiconductors (AlN, GaN, and InN) and their alloys have emerged as promising materials for a wide range of electronic device applications. Aluminum nitride (AlN), which is the widest band gap compound of III-nitride family, exhibits attractive material properties such as wide and direct band gap of 6.2 eV (hexagonal AlN), small (even negative) electron affinity, significant piezoelectric response, good dielectric properties, chemical stability, high thermal conductivity, and low thermal expansion. Owing to this unique set of properties, nanostructures of AlN (e.g., nanotubes,[1–6] hollow nanofibers,[7] etc.) have recently attracted considerable attention as promising candidates for high surface area, high sensitivity chemical and biological sensor applications.[8,9] Template-free or template-based approaches may be adopted for the syntheses of nanostructures. In most cases, template-free methods were reported for tubular AlN nanostructures. Polycrystalline cubic AlN (*c*-AlN) nanotubes were synthesized by gas-phase condensation using the solid–vapor equilibrium and their field emission properties were investigated.[1] Faceted hexagonal AlN (*h*-AlN) nanotubes were obtained by nitriding the aluminum powder in a horizontal tubular furnace.[2] *c*-AlN nanotubes were produced by reacting $AlCl_3$ and NH_3 gases at 1200°C.[3] Nanotubes of AlN were obtained as the side product while synthesizing a bulk layer of AlN polycrystals.[4] Amorphous AlN nanotubes filled with nickel nanoparticles were also realized through the reaction of NH_3 over Ni-Al thin film at 1000°C.[5] Recently, synthesis of *h*-AlN nanotubes by a roll-up approach at 350°C was reported, using AlP and NaN_3 as the Al and N sources, respectively.[6] Polycrystalline *h*-AlN hollow nanofibers were produced by carbothermal reduction and nitridation of precursor fibers obtained by electrospinning.[7]

Methods reported in the above-mentioned studies generally require high temperatures (>1000°C) and in some cases there exist additional morphologies such as nanoparticles or nanowires in the final product. The most obvious constraint of template-free synthesis is probably the limited control over the properties of resulting structure (e.g., crystal structure, dimensions, etc.). Template-based synthesis, on the other hand, is a straightforward way of producing nanostructures with controlled properties, which in general requires a suitable deposition method and a sacrificial substrate having the desired geometry. Selected template material should not only be resistant to the growth ambient (temperature, pressure, gases), but should be able to disappear with a simple postdeposition treatment as well, unless it has a function in the final structure. Yin *et al.*[10] reported on the synthesis of coaxial C-AlN-C composite nanotubes at 1600°C using a chemical substitution reaction in a controllable two-stage process using multiwalled carbon nanotubes as templates. Another example was demonstrated by Stan *et al.*,[11] in which epitaxial *h*-AlN shells were grown by metal-organic chemical vapor deposition at 1000°C around GaN nanowire templates. Templates were then removed by annealing at 1120°C under H_2 atmosphere, leaving behind empty AlN shells.

Temperatures used for the synthesis of AlN nanostructures can be lowered considerably by alternating the deposition method. Atomic layer deposition (ALD) is a special type of low-temperature chemical vapor deposition, in which the substrate is exposed to two or more precursors in a sequential manner. Figure 1(a) is the schematic representation of an ALD cycle, which in general consists of four steps: (i) introduction of the metal-containing precursor, (ii) purge or evacuation, (iii) introduction of the second precursor, and (iv) purge or evacuation. As precursor molecules do not react with themselves, each pulse results in a surface saturated with a monolayer of that precursor.[12] Besides being a low-temperature process, ALD also offers precise thickness control as well as excellent uniformity and conformality with this self-limiting growth mechanism. These unique characteristics make ALD a powerful technique for synthesizing nanostructures through template-based methods.

Variety of templates can be used as the deposition temperature decreases. For instance, polymers are promising
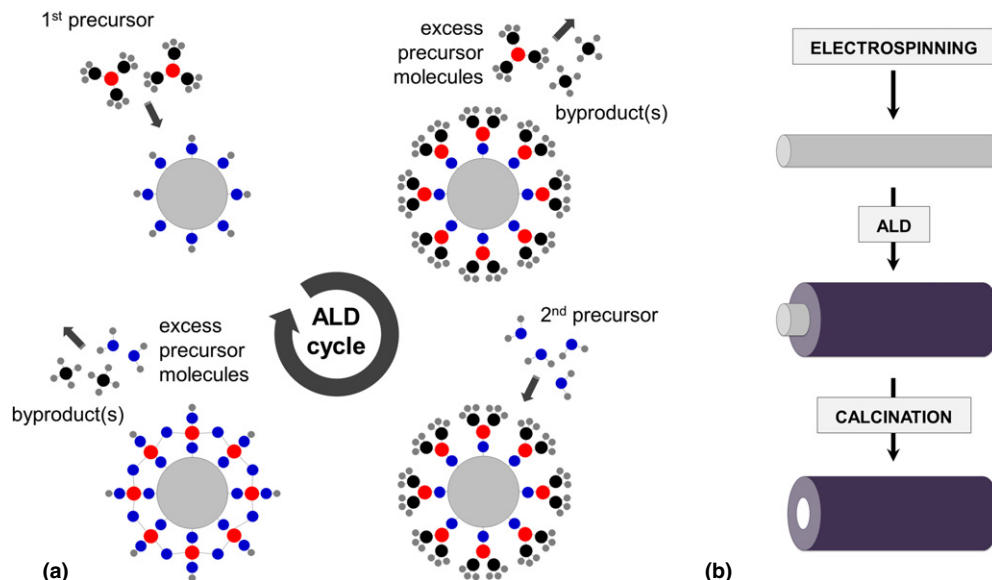
Fig. 1. (a) Schematic representation of an ALD cycle. (b) Template-based synthesis of inorganic hollow nanofibers; preparation of the nanofiber template by electrospinning, conformal deposition on electrospun nanofibers via atomic layer deposition, and removal of the organic template by calcination.

materials as sacrificial templates due to their availability, design flexibility, and very low cost. Polymeric fibers having diameters in the range of few micrometers to few hundred nanometers can be obtained via electrospinning, a basic process in which a polymer solution or melt pumped from syringe is subjected to high voltages. The shapes and dimensions of the electrospun nanofibers can be easily tuned by varying the polymer type, polymer concentration, solvent type, or controlling the electrospinning parameters including applied voltage, tip-to-collector distance, flow rate, etc.[13] Very recently, electrospinning and ALD processes have been combined for synthesizing tubular nanostructures. Peng et al.[14] used electrospun polymeric fiber template for fabricating long and uniform metal-oxide microtubes with precise wall thickness control. In their study, Al_2O_3 was deposited by ALD on electrospun polymeric microfibers; polymeric core was then selectively removed by calcination. This approach was also applied for the fabrication of hollow nanofibers (or nanotubes) of various sizes from various materials such as SnO_2,[15,16] TiO_2,[17,18] and ZnO[19,20] using different electrospun polymeric nanofiber templates. Moreover, $NiFe_2O_4$-TiO_2,[21] TiO_2-ZnO,[22] and SnO_2-ZnO[23] core-shell nanofibers, as well as microtube-in-microtube $ZnAl_2O_4$ assemblies[24] were also fabricated successfully by combining electrospinning and ALD processes. To the best of our knowledge, synthesis of AlN hollow nanostructures by combining electrospinning and ALD has not yet been reported.

Here, we report on the template-based synthesis and characterization of AlN hollow nanofibers. The process has three steps [Fig. 1(b)]: (i) preparation of the Nylon 6,6 nanofiber template by electrospinning, (ii) conformal deposition of AlN on the electrospun polymer template via plasma-enhanced ALD (PEALD), and (iii) removal of the organic template by calcination.

II. Experimental Procedure

(1) Electrospinning of Nylon 6,6
Electrospun Nylon 6,6 nanofiber templates having different average fiber diameters were produced using different solvent systems [1,1,1,3,3,3-hexafluoro-2-propanol (HFIP; ≥99%, Sigma-Aldrich, Chemie Gmbh, Munich, Germany) and formic acid (98%–100%; Sigma-Aldrich, Chemie Gmbh)] and polymer concentrations. 8 wt.% Nylon 6,6 (relative viscosity 230.000–280.000) pellets were dissolved in formic acid; 5 wt.%

and 8 wt.% Nylon 6,6 were dissolved in HFIP separately for 3 h. Prepared homogeneous clear solutions were loaded individually in a 3 mL syringe fitted with a metallic needle having 0.8 mm inner diameter. Syringe was fixed horizontally on the syringe pump (Model: KDS 101; KD Scientific, Inc., Holliston, MA) and polymer solutions were pumped with a feed rate of 1 mL/h during electrospinning. Electrospinning of the solutions was performed by applying a voltage of 15 kV to the metal needle tip by high voltage power supply (AU Series; Matsusada, Precision Inc., Kusatsu-City, Shiga, Japan). Tip-to-collector distance was set at 10 cm. On the way to the grounded stationary cylindrical metal collector (height: 15 cm, diameter: 9 cm) covered by a piece of aluminum foil, the solvents evaporated; and solid electrospun Nylon 6,6 nanofibers were deposited on the collector. The electrospinning setup was enclosed in a Plexiglas box, which allowed electrospinning process to be carried out at 24°C and 30% relative humidity. Morphologies and fiber diameters of the electrospun nanofibers were analyzed by SEM. Average fiber diameters of the samples were calculated by measuring diameters of ~100 different fibers from high magnification SEM images.

(2) Synthesis of AlN Hollow Nanofibers
AlN depositions were carried out at 200°C in Fiji F200-LL ALD reactor (Cambridge Nanotech, Inc., Cambridge, MA) with a base pressure of 30 Pa. Four hundred and 800 cycles of AlN were deposited on electrospun Nylon 6,6 nanofibers via PEALD using trimethylaluminum (TMA) and ammonia (NH_3). One PEALD cycle consisted of 0.1 s TMA/10 s Ar purge/40 s NH_3 plasma (50 sccm, 300 W)/10 s Ar purge. Details of the recipe optimization are given elsewhere.[25] Ar was used as the carrier and purge gas. Precursor and plasma carrier gas flow rates were 60 and 200 sccm, respectively. *In situ* calcination of the AlN-coated nanofibers was carried out at 500°C for 2 h under 260 sccm Ar flow. Samples were taken out from the reactor through a load-lock and exposed to air as soon as the ALD reactor cooled down to 200°C. AlN hollow nanofibers were also prepared by *ex situ* calcination at air ambient (500°C, 2 h).

(3) Characterization Methods
SEM and EDX studies were carried out using Quanta 200 FEG SEM (FEI, Hillsboro, OR) equipped with Ametek

Apollo X silicon drift detector (EDAX Inc., Mahwah, NJ). Samples used for EDX were uncoated, whereas those used for SEM imaging were coated with ~5 nm Au/Pd alloy. TEM and SAED analyses were performed using a Tecnai G2 F30 transmission electron microscope (FEI, Hillsboro, OR). TEM samples were prepared using two different approaches. In the first approach, polymer nanofibers were electrospun directly on copper grids, which were then coated with AlN and calcined *in situ*. Second approach consisted of putting a small piece of AlN hollow nanofiber network into a solvent (i.e., ethanol), followed by sonification to obtain individual hollow nanofibers dispersed through the solvent. AlN hollow nanofiber-containing solvent was then drop casted onto a copper grid and allowed to dry. Chemical composition and bonding states of the AlN nanostructures were investigated by X-ray photoelectron spectroscopy (XPS) using K-Alpha spectrometer (Thermo Fisher Scientific, Waltham, MA) with a monochromatized Al Kα X-ray source.

III. Results and Discussion

Nylon 6,6 nanofiber templates having different average fiber diameters were obtained via electrospinning technique. Characteristics (composition and viscosity) of the Nylon 6,6 solutions, together with the morphologies and average fiber diameters of the electrospun Nylon 6,6 nanofibers are summarized in Table I. Type of the solvent used and concentration of polymer solution affected the polymer solution viscosity that is quite important for fiber diameters. Therefore, the viscosity of each Nylon 6,6 solution was different; accordingly, electrospinning of these solutions yielded Nylon 6,6 nanofibers with different fiber diameters. Less stretching of the electrified jet was occurred for more viscous polymer solutions and larger fiber diameters were obtained from these solutions, as it is anticipated.[26,27] The representative SEM images of uniform and bead-free electrospun Nylon 6,6 nanofiber templates having smooth surfaces obtained from 8% (w/v) formic acid, and 5% and 8% (w/v) HFIP solutions are given in Fig. 2. Average fiber diameters of these Nylon 6,6 nanofibers were measured as ~70, ~330, and ~740 nm, respectively. These randomly oriented Nylon 6,6 nanofibers having different average fiber diameters were used as templates for the fabrication of AlN hollow nanofibers.

Four hundred cycles AlN were deposited on an electrospun template (average fiber diameter ~740 nm) at 200°C by PEALD. Recently, we have reported the deposition rate of AlN at this temperature as 0.86 Å/cycle for planar substrates,[25] which corresponds to a ~34 nm thick film for 400 cycle deposition. As expected, the characteristic self-limiting growth mechanism resulted with highly uniform and conformal AlN layers on electrospun Nylon 6,6 nanofibers [Fig. 3(a)]. However, integrity of these conformal layers could not be retained after *ex situ* calcination at air ambient [Figs. 3(b) and (c)]. Although wall thickness of the resulting inorganic hollow nanofibers could easily be controlled by the number of ALD cycles, there seems to be a critical wall-
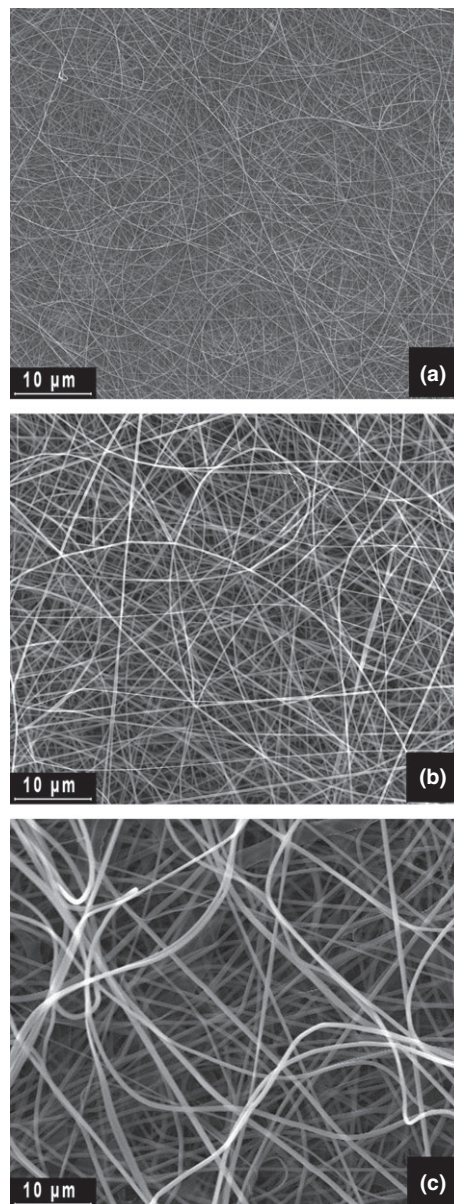

Fig. 2. SEM images of electrospun Nylon 6,6 nanofiber templates having (a) ~70 nm, (b) ~330 nm, and (c) ~740 nm average fiber diameters.

thickness-to-inner-diameter ratio for ALD-grown layers to preserve their shapes after the sacrificial templates have been removed by calcination.

Figure 4(a) shows the SEM image of nanostructures synthesized by 800-cycle ALD growth of AlN (corresponding to ~69 nm AlN wall thickness) on ~740 nm diameter fiber templates followed by an *in situ* heat treatment under continuous Ar flow. Resulting structures were hollow, although they have been calcined at an oxygen-free ambient. The critical wall-thickness-to-inner-diameter ratio could not be reached despite the doubled number of ALD cycles. Conformal AlN layer was unable to preserve the cylindrical geometry of the electrospun fiber template, which resulted in a hollow ribbon-like morphology. Figure 4(b) is the SEM image of hollow nanofibers synthesized by depositing 800 cycles AlN on a template having ~330 nm average fiber diameter. For this combination, the resulting structure was an ideal replicate of the electrospun nanofiber template. EDX indicated the presence of Al, N, O, and C in this sample. SEM image of hollow nanofibers synthesized by depositing 800 cycles AlN on a template having ~70 nm average fiber diameter is given in Fig. 4(c). It is seen that individuality of the fibers has been

Table I. Properties of Nylon 6,6 Solutions and The Resulting Electrospun Nanofibers

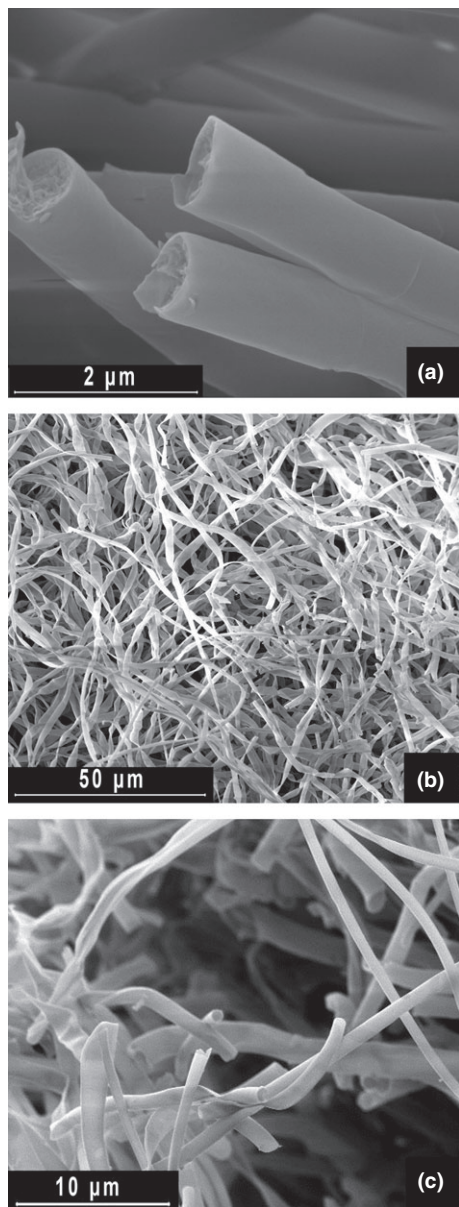| Solvent system	% Nylon 6,6[†] (w/v)	Viscosity (Pa s)	Fiber diameter (nm)	Fiber morphology
Formic acid	8	0.0228	67 ± 35	Bead-free nanofibers
HFIP	5	0.115	330 ± 83	Bead-free nanofibers
HFIP	8	0.24	737 ± 266	Bead-free nanofibers

[†]With respect to the solvent.

Fig. 3. SEM images of (a) Nylon 6,6 nanofibers (average fiber diameter ~740 nm) coated with 400 cycles AlN, and (b, c) inorganic hollow nanofibers synthesized by the *ex situ* calcination of coated nanofibers.
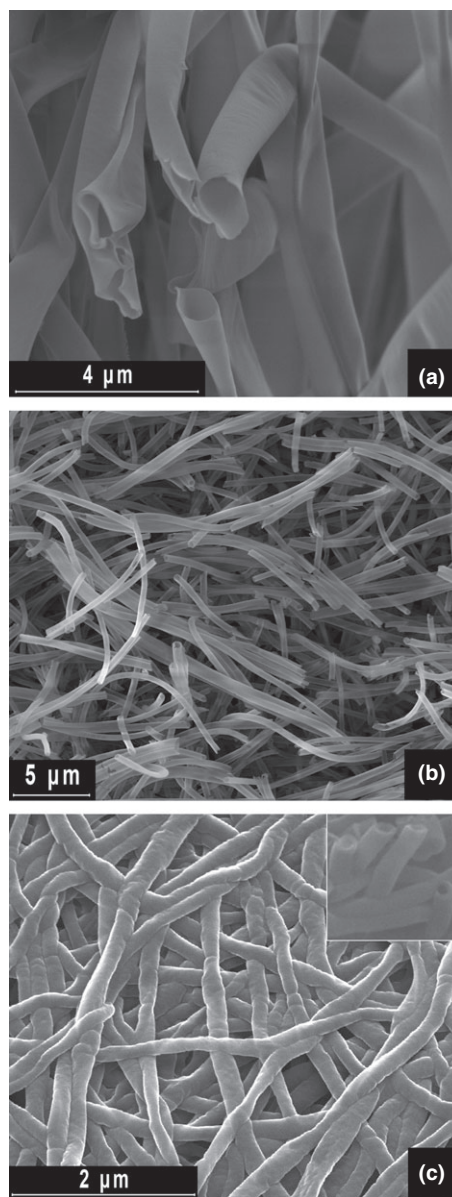

Fig. 4. SEM images of hollow nanofibers synthesized by depositing 800 cycles AlN on Nylon 6,6 templates having (a) ~740 nm, (b) ~330 nm, and (c) ~70 nm average fiber diameters. AlN-coated fibers were calcined *in situ*.

lost due to the coalescence of AlN layers deposited on different fibers. The cross-sectional SEM image of this sample revealed that AlN fibers were hollow [inset of Fig. 4(c)], yet, the wall-thickness-to-inner-diameter ratio for this combination was too high.

Figure 5 belongs to AlN hollow nanofibers synthesized by the deposition of 800 cycles on a template having ~330 nm average fiber diameter, followed by *ex situ* calcination. Each fiber was coated with a uniform and conformal layer of AlN. These layers preserved their shapes even after calcination at air ambient and resulted in a structure that is composed of continuous hollow nanofibers.

Bright field scanning TEM image of sample prepared by the deposition of 800 cycles on a template having ~70 nm average fiber diameter, followed by *in situ* calcination is given in Fig. 6(a). It is seen that the wall thicknesses of hollow nanofibers are highly uniform along the fiber axes. Figures 6(b)–(d) are the TEM images of samples prepared using templates having average fiber diameters of ~70, ~330, and ~740 nm, respectively. Wall thicknesses of the AlN hol-

low nanofibers shown in Figs. 6(b) and (c) were measured as ~65 nm, which is quite consistent with the deposition rate of AlN PEALD process at 200°C. For the sample shown in Fig. 6(d), wall thickness was ~32 nm. This value is lower than the expected coating thickness, and might be related to the TEM sample preparation approach that was followed. For the samples shown in Figs. 6(a)–(c), few nanofibers were electrospun on copper grids, which were then coated and calcined *in situ*. These fibers were at the very top during the PEALD of AlN. For the sample shown in Fig. 6(d), on the other hand, TEM sample was prepared by sonication and drop casting. This individual nanofiber might therefore belong to any position along the out of plane direction. As AlN recipe has been optimized for planar substrates, the precursor doses used in this experiment may not be enough for coating such a high surface area sample. Even if the TMA and NH_3 doses are sufficient for self-terminating reactions to take place at the fiber surface, there may have not been enough time for them to diffuse into the nanofiber matrix.

Crystal structure of the AlN film deposited on Nylon 6,6 nanofiber templates was studied by high-resolution TEM (HR-TEM) and SAED. HR-TEM image of the AlN hollow nanofiber synthesized using ~330 nm diameter Nylon 6,6 nanofibers is given in Fig. 6(e). AlN was found to be poly-crystalline wi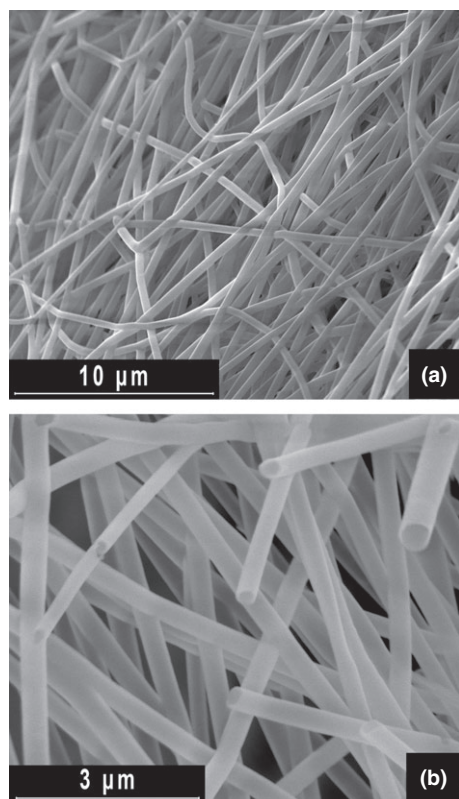th nanometer-sized grains, which was further confirmed by SAED. SAED pattern of the same sample [Fig. 6(e)] revealed seven polycrystalline diffraction rings, representing the *h*-AlN phase. Table II summarizes the SAED results, theoretical values for *h*-AlN, and corresponding planes. When same recipe was deposited on planar Si (100) substrates, the same seven reflections of the *h*-AlN phase appeared in the grazing-incidence X-ray diffraction pattern.[25]

Chemical compositions of bare and coated Nylon 6,6 nanofibers, as well as synthesized AlN hollow nanofibers were investigated by XPS, which is a surface analysis technique that collects data from the top ~5–10 nm of the sample. In this study, the relatively large spot size (~400 μm) of the X-ray beam interacted with a large number of coated or calcined nanofibers, which might have discontinuities or cracks on the AlN shell due to the sample preparation procedure applied. Therefore, the collected data probably represent the organic content and Nylon 6,6–AlN interface in addition to the PEALD-grown AlN surface. Survey scans detected peaks of Al, N, O, and C for coated and calcined samples prepared by depositing 800 cycles AlN on a template having ~330 nm average fiber diameter (Table III). *Ex situ* calcination at 500°C for 2 h must have resulted in complete removal of the organic component. Accordingly, Al content increased and N content decreased when coated sample was calcined. An increased amount of O is believed to be due to the oxidation of AlN films upon annealing at air ambient. The 19.18 at.% C present in the calcined sample, on the other hand, corresponds either to the surface contamination or calcination residues. XPS survey scan results of *in situ* calcined sample were quite similar to those of coated sample. There was no significant decrease in C concentration upon calcination. In other words, calcination at an oxygen-free ambient was not as effective as that performed at air ambient, although SEM images showed hollow structures for both cases. Results therefore indicate oxidation of AlN layer in the case of *ex situ* calcination and ineffectiveness of *in situ* calcination in terms of removing the organic component.

High-resolution XPS (HR-XPS) scans were also obtained to reveal bonding states of AlN hollow nanofibers synthesized at different conditions. Charging effects were corrected
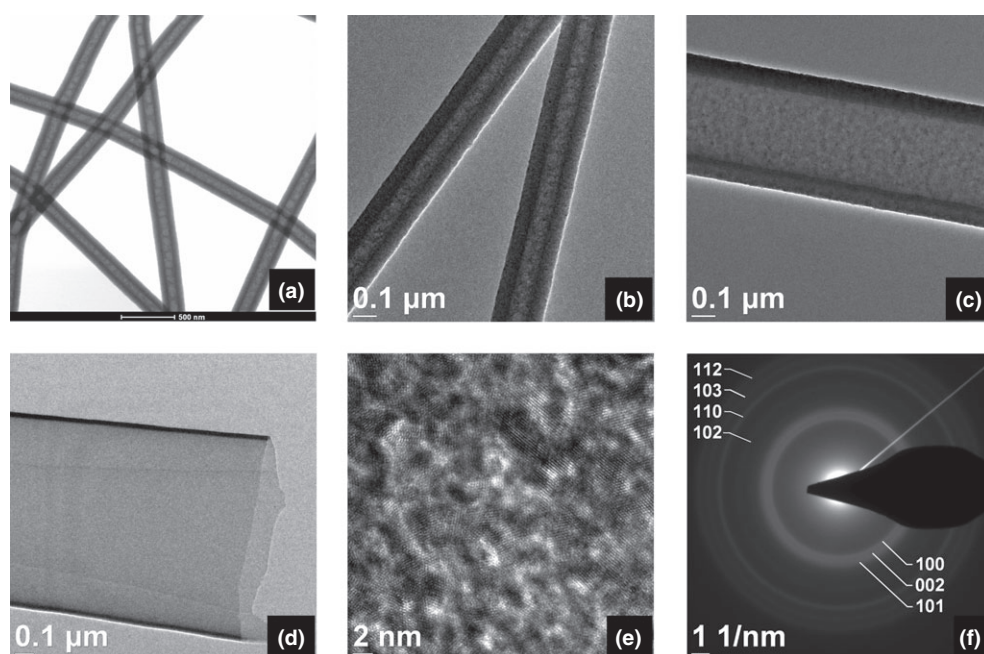

Fig. 5. SEM images of AlN hollow nanofibers taken at different magnifications. Hollow nanofibers were synthesized by the deposition of 800 cycles AlN on a Nylon 6,6 template having ~330 nm average fiber diameter, followed by *ex situ* calcination at air ambient.

Fig. 6. AlN hollow nanofibers synthesized by the deposition of 800 cycles AlN, followed by *in situ* calcination. (a) Bright field TEM image of hollow nanofibers synthesized using Nylon 6,6 template having an average fiber diameter of ~70 nm. (b)–(d) TEM images of hollow nanofibers synthesized using templates with average fiber diameters of ~70, ~330, and ~740 nm, respectively. (e) High-resolution TEM image, and (f) SAED pattern of AlN hollow nanofibers synthesized using a template having ~330 nm average fiber diameter.

Table II. SAED Results, Theoretical Values, and Corresponding Crystallographic Planes

Diameter (1/nm)	Interplanar spacing, d (Å)		Corresponding plane, hkl
	Calculated	Theoretical[†]	
7.434	2.690	2.6950	100
8.057	2.482	2.4900	002
8.451	2.367	2.3710	101
11.036	1.8123	1.8290	102
12.952	1.5442	1.5559	110
14.199	1.4085	1.4133	103
15.261	1.3105	1.3194	112

[†]Hexagonal AlN, ICDD reference code: 00-025-1133.

Table III. XPS Survey Scan Results. 800 Cycles AlN Were Deposited on a Template Having ~330 nm Average Fiber Diameter

Sample	Elemental composition (at.%)			
	Al	N	O	C
Bare template	–	9.79	11.71	78.5
Coated template	20.69	8.33	33.74	37.24
After *ex situ* calcination	33.02	5.78	42.02	19.18
After *in situ* calcination	21.87	7.51	37.14	33.49

for coated and calcined samples using the adventitious carbon peak located at 285 eV. C 1s peak of the bare template having ~70 nm average fiber diameter was fitted by multiple subpeaks, one of which (corresponding to the surface C contamination) was found to be located at ~285 eV. No correction was therefore made for the bare template. Al 2p HR-XPS scans of hollow nanofibers synthesized by the *in situ* calcination of templates coated with 800 cycles AlN are shown in Fig. 7(a). As expected, Al 2p peaks were found to locate at the same position for samples synthesized using templates having different average fiber diameters (i.e., ~70 and ~740 nm). The data obtained from the sample prepared using ~740 nm average fiber diameter template was fitted by two subpeaks located at 73.33 and 74.32 eV, corresponding to Al–N[28] and Al–O[29] bonds, respectively. The Al–O bond indicates surface oxidation, which might be expected as the samples were taken out from the ALD reactor and exposed to air at 200°C. N 1s HR-XPS scans of the samples, as well as that of the bare template having an average fiber diameter of ~70 nm are given in Fig. 7(b). N 1s data obtained from the sample prepared using ~740 nm average fiber diameter template were fitted by two subpeaks. The peak located at 396.57 eV was attributed to the N–Al bond[28]; whereas the one located at 399.06 eV was assigned as the N–O bond.[30] The presence of the N–O bond, which was also observed for the bare Nylon 6,6 template, is a direct proof of the existence of organic component in the sample prepared by *in situ* calcination. The peak representing the N–O bond was also observed for the sample coated with 800 AlN cycles; however, it disappeared after *ex situ* calcination at air ambient. The temperatures required for the removal of sacrificial Nylon 6,6 nanofiber templates are not high and the whole process can be carried out inside the ALD reactor. As Nylon 6,6 nanofibers could not be completely removed through heat treatment at an oxygen-free ambient, the gas composition and gas flow rates must be adjusted accordingly during calcination.

Al 2p HR-XPS scans of coated and calcined (both *in situ* and *ex situ*) samples prepared using polymeric templates having ~70 nm average fiber diameter were all fitted by two subpeaks located at 73.5 ± 0.2 (Al–N bond) and 74.5 ± 0.3 eV
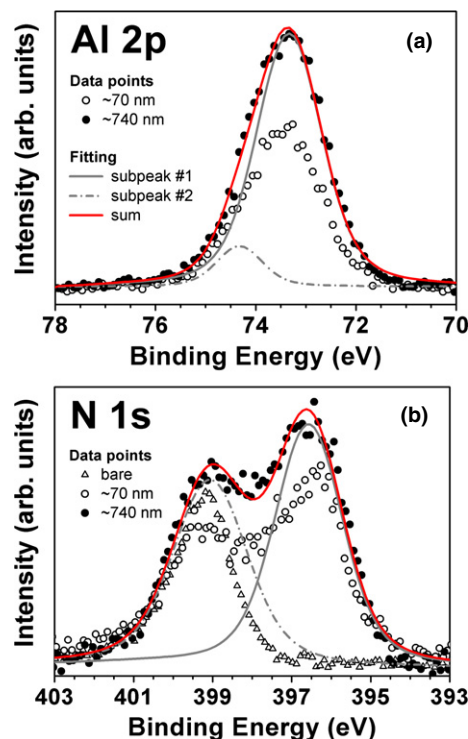

Fig. 7. (a) Al 2p and (b) N 1s high-resolution XPS scans of AlN hollow nanofibers synthesized by depositing 800 cycles AlN on Nylon 6,6 templates having ~70 and ~740 nm average fiber diameters, which was followed by *in situ* calcination. N 1s high-resolution XPS scan of bare Nylon 6,6 nanofiber template having an average fiber diameter of ~70 nm is also included.
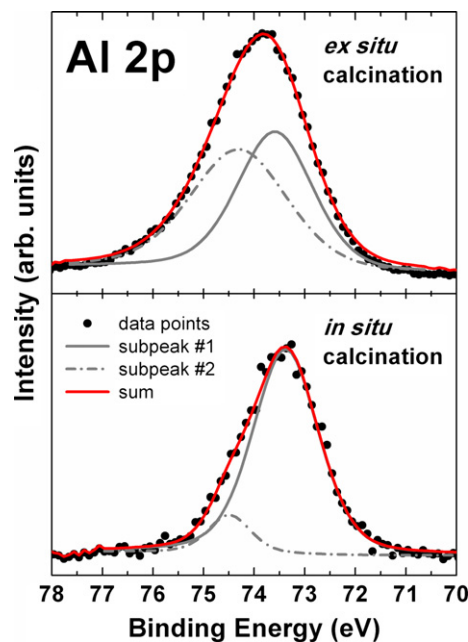

Fig. 8. Al 2p high-resolution XPS scans of AlN hollow nanofibers synthesized by the deposition of 800 cycles AlN on Nylon 6,6 nanofiber templates (average fiber diameter ~70 nm), followed by *in situ* and *ex situ* calcinations.

(Al–O bond). Figure 8 shows the Al 2p HR-XPS scans of calcined samples. When compared with that prepared by *ex situ* calcination, the sample prepared by *in situ* calcination exhibited a lower Al–O/Al–N subpeak area ratio (0.18 vs 0.86) as expected.

IV. Conclusions

Template-based synthesis of AlN hollow nanofibers was demonstrated by combining the electrospinning and PEALD processes. Conformal AlN films were deposited at 200°C on sacrificial electrospun Nylon 6,6 nanofiber templates, using TMA and NH_3 plasma. AlN-coated nanofibers were then calcined *in situ* or *ex situ* at 500°C for 2 h to remove organic components. SEM studies showed that there is a critical wall-thickness-to-inner-diameter ratio for AlN hollow nanofibers to preserve their shapes after the polymeric template has been removed by calcination. TEM images indicated uniform wall thicknesses along the fiber axes. Synthesized AlN hollow nanofibers were polycrystalline with a hexagonal crystal structure as determined by HR-TEM and SAED. Such high surface area AlN hollow nanofibers might potentially be used in high temperature ambient chemical sensing applications, where both high temperature compatibility and high sensitivity should meet.

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