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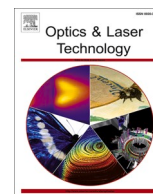
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Full length article

High-temperature resistant boron nitride-based coatings for specialty silica optical fibers

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ABSTRACT

h-BN is well known for its thermal stability. *h*-BN coated silica optical fibers were manufactured. The coating solution is compatible with optical fiber drawing process, which allows the preparation of tens of meter of *h*-BN coated fibers. In addition, it is possible to use this coating material for the preparation of short length fiber samples in a post-production approach. Bentonite acts as a binder and allows the adhesion of *h*-BN to the silica optical fiber. The thermal stability of this protected optical fiber has been demonstrated over a short period of time (several hours) up to 900 °C and over a long period of time (1500 h) up to 800 °C in air. Moreover, the coated optical fiber can resist up to 1000 °C in neutral atmosphere (tested over 6 h).

1. Introduction

Silica optical fibers are used in a wide variety of high-tech areas such as telecommunications, medical, instrumentation and so on [1,2]. Their manufacturing processes are continuously optimized to meet requirements needed by each application. From a general manufacturing point of view, Outside Vapor Deposition [3], and Modified Chemical Vapor Deposition [4] are the two main processes for the fabrication of silica optical preforms. These preforms are then transformed into fibers using a fiber drawing tower. During the process, micro-cracks remain on the silica surface. These defaults must be protected against air atmosphere attack. A prolonged exposure to air will cause the fiber to break. Moreover, the silica optical fiber must be mechanically protected. In this aim, a protective coating is added at the surface of the glass clad as shown in Fig. 1 [5].

Technically, the silica optical fiber is coated during the drawing process before being wound onto the drum [6] (Fig. 2).

Historically, organic polymers, such as acrylate or polyimide, are the main sources of coating for silica optical fiber [7,8]. These coatings are relevant for telecommunication applications. Recent developments of optical fiber sensors have demonstrated that optical fibers are promising candidates for strain and temperature monitoring: compactness, reliability and electromagnetically immunity, of optical fiber are

particularly of interest in aeronautic and nuclear domain [9,10]. Indeed, in these fields, optical fiber sensors appear as promising candidates for sensing and distributed temperature/strain measurements without electromagnetic interference or electric signal. Additionally, silica or sapphire fibers withstand temperatures up to 1000–1200 °C [11,12]. However, the thermal stability of standard coatings suffers limitation. The short-term temperature resistance of polyimide coating for instance is 400 °C and the long-term use temperature range is 200–300 °C [5,13,14]. Recently, polyamide-imide polymers have demonstrated their ability to protect the fiber up to 350 °C for 72 h [15]. Metal coatings such as silver, aluminium, or gold, are considered for harsh environment application, regarding their greater melting point greater than organic polymers. Aluminium and gold-coated fibers are already commercially available. Post process silver coating of silica fiber is also achievable on small length, by a Tollens reaction for instance [16,17]. In all the above cases, metal-coated fibers can't work on long period of time at temperature in excess of 400 °C, while many applications (aircraft engine or nuclear reactor monitoring) require operating temperature beyond 600 °C.

In this context, the development of optical fiber coatings that can withstand temperature beyond 400 °C remains a technological challenge. In addition, one can note that fiber length to be coated depends on the kind of sensor: The length parameter will drive the deposit process to

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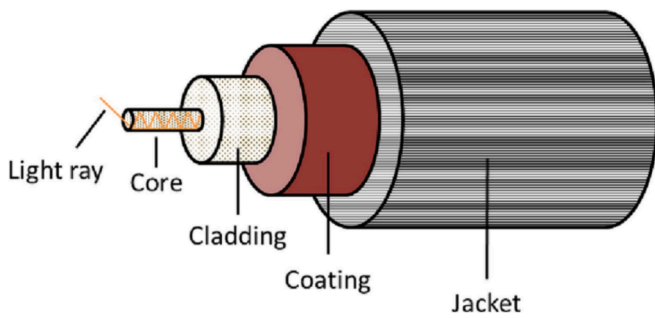


Fig. 1. Scheme of optical fiber structure [5].

be adopted. Manufacturing of long length of silica optical fiber coated with a high temperature material (up to 500 m) would need to coat the fiber directly during the fiber drawing process, whereas small length samples (up to 50 mm for example) would require post deposition process [18]. Thanks to their high melting point, resistance to oxidation and chemical stability, advanced ceramics are the best candidates to solve this issue. In fact, hexagonal boron nitride, *h*-BN, presents a thermal stability up to 800–900 °C in air [19]. Moreover, *h*-BN presents a good resistance to thermal shock. Its structure, similar to that of graphite, makes it a suitable material for optical fiber coating compared with other ceramics such as titanium oxide (TiO₂), zirconium oxide (ZrO₂) and aluminium oxide (Al₂O₃) for long-term high-temperature resistant sensors [20,21].

Manufacturing fiber sensor with *h*-BN coating on silica fiber was demonstrated on small length sample by Luan et al. [22]. In their work, the *h*-BN is sputtered on the fiber by a Chemical Vapor Deposition

process. Thermal stability of a coated fiber was demonstrated up to 700 °C. The thickness of the *h*-BN film [0.5–2.85 μm] increases with the deposition time [5–30 h]. However, the process and the duration of the deposition is not compatible with the fiber drawing process [several meter/minutes].

In this article, we report the preparation of a ceramic coating based on boron nitride and its deposition on silica optical fibers as protective coating. In the process, Bentonite, bento, is mixed to *h*-BN act as a binder. The deposition of the ceramic coating is compatible with fiber drawing system and thus can be applied during the manufacturing of the optical fiber. The manufacturing of more than 100 m long optical fiber covered by this ceramic coating is presented. It is also possible to apply this coating in a post-process approach over short length of optical fiber (50–2500 mm in length) using a home-made device.

The thickness of the *h*-BN film is about 10 μm when applied during the silica optical fiber drawing. In post-process approach, the thickness can be tailored from 10 μm to 200 μm typically. Chemical and physical characterizations such as Differential Scanning Calorimetry (DSC), ThermoGravimetric Analysis (TGA), X-Ray Diffraction (XRD) and optical microscopy confirm the high thermal stability in air up to 900 °C (short-time) and 800 °C (long time, tests made during up to 1500 h): neither crack formation nor chemical modifications of the coating have been observed. Thermal Treatment made under argon (Ar) have also shown the thermal stability of *h*-BN@bento coating up to 1000 °C.

2. Material and methods

Bentonite, Bento, and hexagonal Boron nitride, *h*-BN, were purchased from Sigma-Aldrich supplier (France) and used without any further purification. Milli-Q water with a resistivity of 18.2 MΩ cm was used in

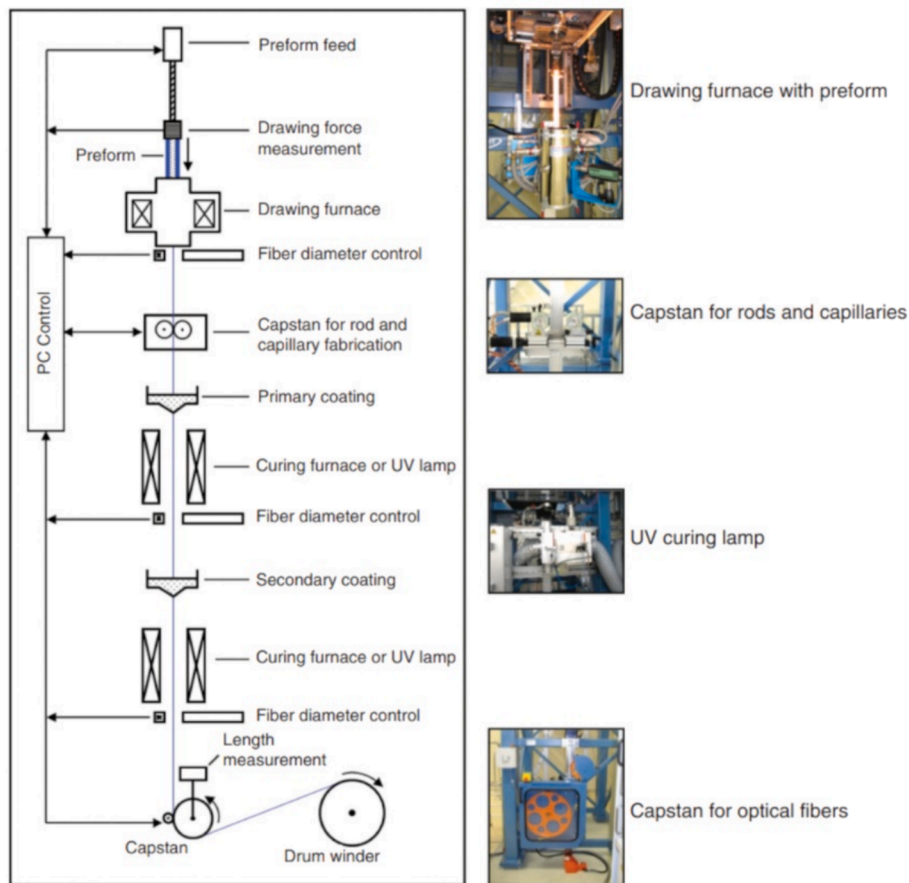


Fig. 2. Conventional drawing tower setup [6].

all the preparations. Optical loss was recorded using an Optical Time Domain Reflectometer (OTDR) JDSU MTS8000. Microscopy images are recorded on a Nikon eclipse LV100. XRD diagrams were recorded with a Bruker AXS D8 diffractometer using a Cu K_{α} radiation source (40 kV and 40 mA) and equipped with a Lynxeye XE 1D detector. Experiments were carried out between $2\theta = 10^{\circ}$ and 60° on powder, in $\theta/2\theta$ configuration. A TA Balance instrument, model: SDT 2960 DSC-TGA X, was used to conduct thermal analysis (TGA/DSC) under an atmosphere of pure air or nitrogen (N_2) ($100 \text{ mL}\cdot\text{min}^{-1}$). The sample was heated at a rate of $10^{\circ}\text{C}/\text{min}$ from room temperature (RT) to 1200°C . Samples of about 30 mg were introduced into a small corundum crucible; an empty crucible was used as reference for thermal analysis. Specific surface areas of the solid materials were measured by low-temperature nitrogen adsorption at -196°C on a Micromeritics Flowsorb 3 setup using a standard BET procedure. Before analysis samples of about 400 mg were degassed at 120°C for 30 min under a mixture of 30 % N_2/He .

3. Results-discussion

3.1. Preparation of *h*-BN@bento coated optical fibers

The coating material was prepared by adding bentonite to *h*-BN powder. Bentonite, thanks to its hydrophilic properties acts as a binder. Preliminary results showed that below 10 wt% of bentonite, *h*-BN does not adhere to the fiber. On the other hand, when the mass percentage of bentonite is greater than 35 wt%, the coated optical fiber becomes very brittle and rigid. Optimized mixture of *h*-BN containing 12 wt% of bentonite was dispersed in deionised water until obtaining a homogeneous white paste, *h*-BN@bento.

Optical fibers were drawn using a 12 m-high optical fiber draw tower, starting from Heraeus F300 glass or preform produced via the MCVD method. *h*-BN@bento is deposited by using dies similar to those used with conventional polymers coating. The coating layer is deposited under static conditions [23]. Drawing parameters: Preform feed: 7,8 mm/min; Drawing furnace temperature: 1870°C ; Primary-coating: *h*-BN@bento; Curing furnace temperature: 360°C ; Capstan feed: 7 m/min; fiber tension: 110 g; no secondary coating. The drawing speed can be further increased by increasing the curing furnace length. With this process, several tens of meters of standard silica optical fibers were successfully coated, with ceramic layer of about $10 \mu\text{m}$ in radial thickness, on a 1 m circumference drum.

3.2. Optical properties

Attenuation measurements were performed on SMF-like optical fibers coated with $10 \mu\text{m}$ of *h*-BN@bento with an OTDR (Fig. 3). The core is constituted by germanium doped silica ($8 \mu\text{m}$ diameter). Optical cladding is high purity fused silica ($128 \mu\text{m}$ diameter), Δn value is 0.006.

The optical loss level is compatible with the realization of fiber sensors, where maximum fiber lengths are about few tens of meters. This loss level could be reduced by increasing the fiber drawing speed for instance, in order to get closer to the parameters used for telecom optical fiber manufacturing (several tens of m/min). This could be achieved with a taller tower and a longer curing oven.

3.3. Physical properties of coating

During the manufacture of the fiber, the thickness of *h*-BN@bento coating is about $10 \mu\text{m}$ and is homogenous throughout the length of the fiber. The diameter of the core is about $8 \mu\text{m}$. The diameter of the cladding is about $128 \mu\text{m}$. In post-process, on short samples of fiber up to 2.5 m length, the thickness of the coating can be increased up to $200 \mu\text{m}$ in post process (that is to say outside of the drawing tower) by successive multi-layer deposition, using a home-made device [24]. The thickness of the coating is increased by successive application of coating layer, dried at 100°C between each layers (Fig. 4).

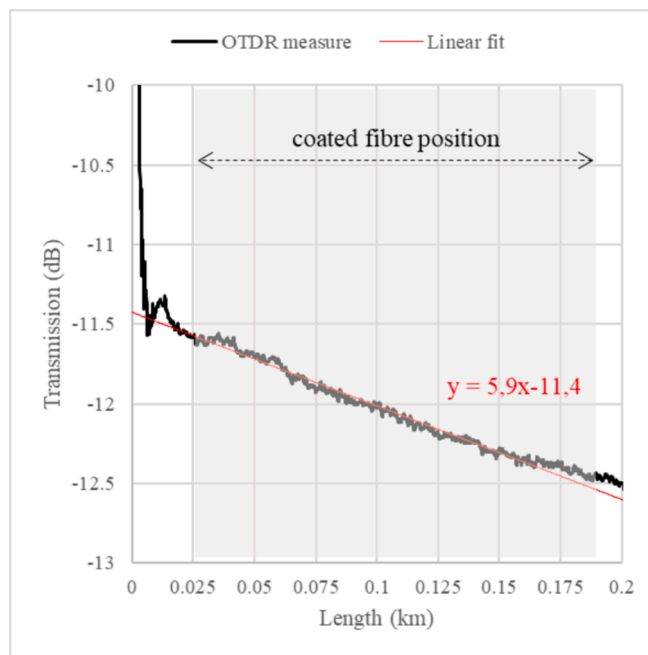


Fig. 3. Optical loss in standard single-mode optical fiber at 1550 nm.

3.4. Thermal stability in air and neutral atmosphere

Silica fiber samples (core diameter: $7 \mu\text{m}$; cladding diameter: $125 \mu\text{m}$) of 40 cm in length, coated by *h*-BN@bento (thickness of about $50 \mu\text{m}$) were heated in a furnace in air or in Ar at 700°C , 800°C , 900°C , and 1000°C . The conditions of tests are presented in Table 1.

No coating degradation (neither cracks nor thickness changes) was observed at 800°C , even after heating during 1500 h (Fig. 5).

Progressive and slow decreasing of the roughness of the coating surfaces were observed when increasing the temperature at 900°C in air. But no crack or delamination were observed in any of the samples. Heat treatments at 900°C and 1000°C in air and in Ar for 6 h were performed to confirm the slow degradation of the *h*-BN in air.

In order to explain the visual observations, XRD analyses were carried out (Fig. 6). Diagrams were recorded on powder after heat treatment at 700°C , 800°C , 900°C and 1000°C in air. Due to its low quantity, peaks of bentonite appear with low intensity. Moreover, as bentonite is composed by several phases that change with temperature, montmorillonite structure turns to amorphous state at 700°C thus, peaks at $2\theta = 20^{\circ}$ and 35° disappeared on diagrams [25–28]. One can observe, on each diagram, that the characteristic peaks of *h*-BN crystal structure are at $2\theta = 26.75^{\circ}$ (002), 41.61° (010), 43.82° (011), 50.16° (012) and 55.11° (004) (ICCD card PDF 00-034-0421) (Fig. 6 (a)).

Diagrams recorded after thermal treatments at 900°C and 1000°C show additional peaks at $2\theta = 14.5^{\circ}$ – 14.9° (1 -1 0)(1 0 0)(broad band), and 28.05° (002) attributed to $B(\text{OH})_3$ (ICCD card PDF 01-073-2158). The progressive oxidation of *h*-BN explains what have been observed on the microscopy images. Moreover, it is in good agreement with the literature. Indeed, it was demonstrated that *h*-BN turns onto boron oxide at high temperature [29,30], and depending on the relative humidity, boron hydroxide $B(\text{OH})_3$ can be obtained [31,32]. The relative low intensity of these peaks compared to those of *h*-BN shows that the transformation takes place over a long period of time. Garo et al. studied the kinetic of this oxidation. They have shown that the transformation takes place up to 50 h with a decrease of the oxidation effect with time. This point also demonstrates that *h*-BN@bento coating can resist at 900°C over a short period of time.

Oxidation of *h*-BN was confirmed by additional analysis after treatment at 1000°C in Ar (Fig. 6 (b)). No degradation of *h*-BN and *h*-

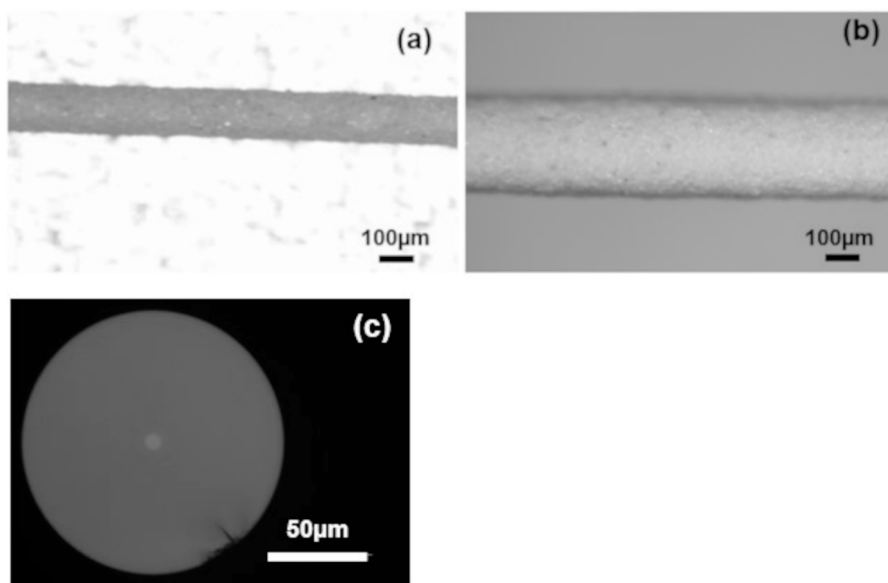


Fig. 4. Microscopy images of (a) standard single-mode silica fiber coated with h-BN@BENTO manufactured by drawing fiber system, (b) after three layers, (c) section of optical fiber without coating.

Table 1
Conditions of thermal treatments.

Sample	Temperature (h)	Duration (h)	Atmosphere
1	700	1500	Air
2	800	1500	Air
3	900	6	Air
4	1000	6	Air
5	900	6	Ar
6	1000	6	Ar

BN@bento was observed in Ar. The peak observed at $2\theta = 21.3^\circ$ was attributed to bentonite transformation.

Differential Scanning Calorimetry (DSC) is performed on powder to confirm the stability up to 1100 °C under N₂ atmosphere and up to 900 °C in air (Fig. 7).

Thermal analysis of h-BN@bento confirms the slow oxidation of h-BN

at 900 °C. As shown in Fig. 7, curve c, the observed mass gain is consistent with molar mass difference between h-BN and B(OH)₃. When the analysis is done under N₂, no significant mass change was observed up to 1200 °C (Fig. 7 curve b). The exothermic peak observed by DSC analysis in air from 900 °C is coherent with the weight gain and with the oxidation of h-BN. These results are also observed on h-BN sample (Fig. 8).

3.5. Textural properties

Textural properties of the solids are listed in Table 2.

The results show that the surface area remains constant from room temperature to after calcination at 800 °C in air or after heat treatment at 1000 °C in Ar. This is consistent with the fact that the coating does not change up to this temperature range. One can observe that for calcined powder at 900 °C and 1000 °C, a decrease of the surface area value to 1.2

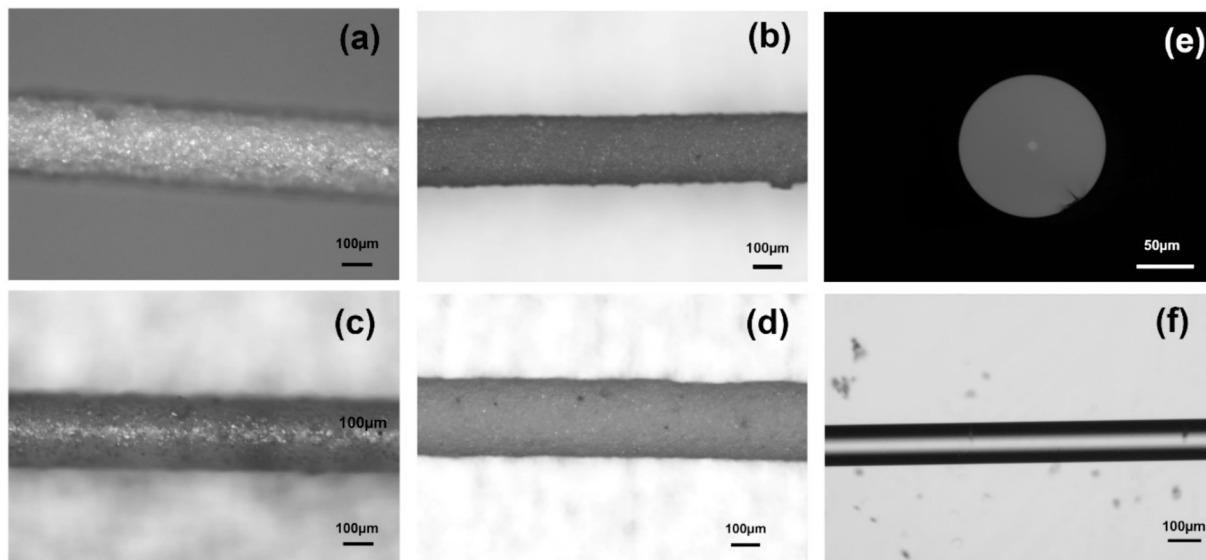


Fig. 5. Coating surface (a) after 800 °C cycles in air (1500 h), (b) after 6 h at 900 °C in air, (c) after 6 h at 1000 °C in air, (d) after 6 h at 1000 °C in Ar, (e) cross section of uncoated optical fiber and (f) uncoated optical fiber.

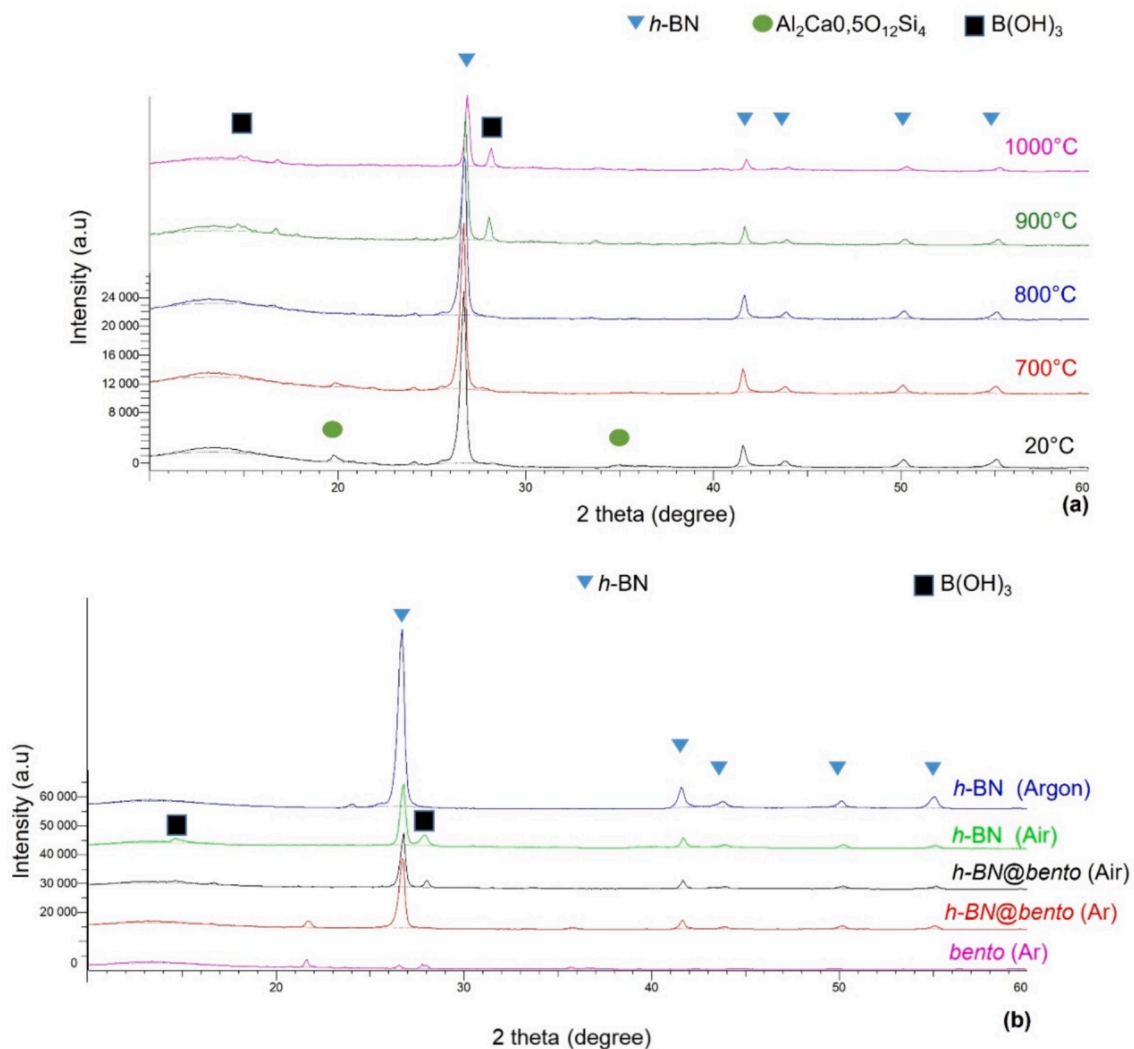


Fig. 6. XRD diagrams of (a) *h*-BN@bento after heat treatments for 12 h in air at 700 °C, 800 °C, 900 °C and 1000 °C, (b) XRD diffractograms of *h*-BN@bento, *h*-BN and bento after heat treatment for 6 h at 1000 °C in air and Ar.

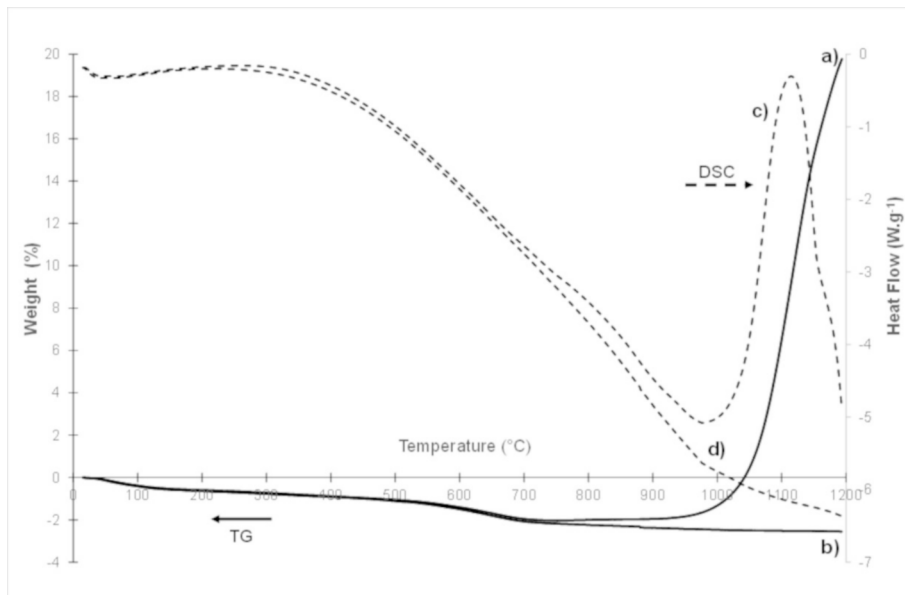


Fig. 7. TGA/DSC Analysis of *h*-BN@Bento: Weight (%) (a) in air (b) in N₂ and Heat Flow (W.g⁻¹) (c) in air (d) in N₂.

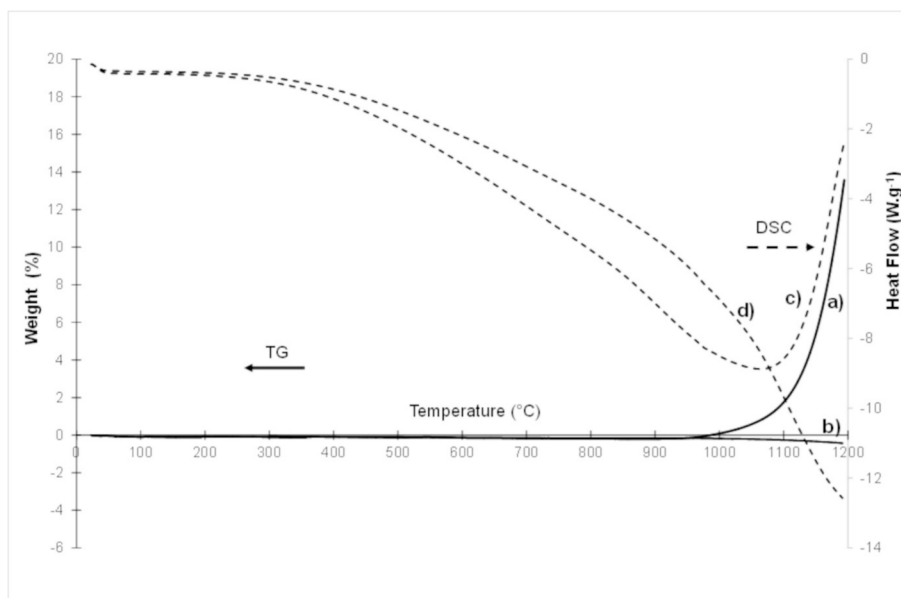


Fig. 8. TGA/DSC Analysis of *h*-BN: Weight (%) (a) in air (b) in N_2 and Heat Flow ($W.g^{-1}$) (c) in air (d) in N_2 .

Table 2

Surface area of solids.

Solid	BET surface area ($m^2.g^{-1}$)
<i>h</i> -BN@Bento at room temperature	15.4
<i>h</i> -BN@Bento calcined 12 h 800 °C air	11.4
<i>h</i> -BN@Bento calcined 12 h 900 °C air	1.2
<i>h</i> -BN@Bento calcined 12 h 1000 °C air	0.7
<i>h</i> -BN@Bento calcined 6 h 1000 °C Ar	12.5

and $0.7 m^2.g^{-1}$ respectively, that could be explained by the progressive formation of $B(OH)_3$ (see).

4. Conclusions

In this paper, silica optical fibers were successfully coated with *h*-BN@bento directly applied during the fiber drawing process with fabricated fiber lengths reaching several tens of meters. The coating's thickness is about 10 μm . This ceramic coating can also be deposited onto silica optical fiber in post process on sample up to 2.5 m in length thank to a home made device. In this case, the coating's thickness can be tuned by multi-layer deposition technique (up to 200 μm demonstrated). The thermal behaviour of the coating has been investigated, on material only at first, and then after deposition on silica optical fiber. Long-term thermal stability of the coating was demonstrated up to 800 °C and for 1500 h: no physicochemical degradation has been observed. Moreover, short time stability up to 1000 °C (several hours) was also shown. In a near future, the *h*-BN@bento coating will be used to realize high temperature optical fibre sensors for harsh environment monitoring.

CRedit authorship contribution statement

Rémy Bernard: Writing – review & editing, Writing – original draft, Supervision, Investigation, Conceptualization. **Aymeric Pastre:** Writing – review & editing, Investigation. **Anne Griboval:** Investigation. **Vincent Andrieux:** Investigation. **Kilian Técher:** Investigation. **Guillaume Laffont:** Writing – review & editing, Funding acquisition. **Laure Lagorached:** Writing – review & editing, Validation, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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