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Boron nitride by pyrolysis of the melt-processable poly[tris(methylamino)borane]: Structure, composition and oxidation resistance

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Abstract

Evolution of structure and composition of the melt-processable poly[tris(methylamino)borane] (PTMB) during its conversion to ceramics was studied by TGA, FTIR, Raman, XRD, XPS and elemental analysis (EA). The results show that the ceramic yield was greatly improved from 60.22 to 74.4 wt% at 900 °C by curing in NH_3 prior to pyrolysis. The carbon impurity in the precursor can be removed effectively in NH_3 whereas no similar result occurred in N_2. In NH_3, 93 wt% of carbon was removed at 600 °C and the carbon content in the pyrolyzed product at 900 °C was only 0.37 wt%. At the same time, the conversion from polymer to ceramics was almost completed at 900 °C. Moreover, the sample acquired at 900 °C was amorphous boron nitride (BN), while that of further annealing at 1600 °C showed characteristic of turbostratic BN (*t*-BN). Additionally, the BN with nearly stoichiometric composition exhibited good oxidation resistance even up to 900 °C in air.

Keywords: Preceramic polymer; Pyrolysis; Boron nitride; Structure; Oxidation resistance

1. Introduction

Nonoxide ceramics derived from preceramic polymers, such as polycarbosilane-derived SiC [1,2], polyborazine-derived BN [3,4] and polyborosilazane-derived SiBN(C) [5,6] have all been researched intensively in recent years. Among them, BN has received great attention as a result of its high chemical stability, low density, excellent oxidation resistance and lubricant characteristic, etc. Nevertheless, it is not easy to fabricate BN with complex shapes using traditional powder sintering and high temperature nitruration methods [7,8]. Fortunately, the polymer-derived ceramics (PDCs) route provides a good control of the preceramic polymer's processability to fabricate near-net shapes in a way not known from other techniques [9,10].

Up to date, polymeric precursors to shaped BN were based on borazine [11], B-trichloroborazine [3,12] and (alkylamino)borane monomers [13]. As one of the easily synthesized polymeric precursors to BN, poly[tris(methyalmino)borane] (PTMB) exhibited great potential in fabricating complex BN devices, such as fibers and coatings [13–19]. As far as we know, though pyrolysis of polyborazene [20] and poly[(alkylamino)-borazine] [21–23] has been widely investigated, that of poly[alkylamino)borane], especially for PTMB, have received less attention. In the present research, evolution of structure and composition of PTMB and anti-oxidation property of BN derived therefrom were studied.

2. Experimental

All samples described in this work were manipulated in a dry Ar atmosphere. The PTMB precursor was synthesized as reported previously [13,24], which contains the synthesis of tris(methylamino)borane monomer and followed thermal condensation. In a pyrolysis procedure, the polymer precursor lumps were firstly ground to a fine powder with a diameter less than 20 μm in a glove box, and then cured in NH_3 for a while. After that, the cured powders were weighed into an alumina

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boat and then placed in the pyrolysis tube. After purging the system with Ar for 30 min, the sample was heated to the final temperature (4 °C/min) under flowing anhydrous NH_3 (80 ml/min) with a holding time of 2 h. It was then cooled to room temperature naturally. Moreover, the pyrolysis product obtained at 900 °C was annealed for 1 h at temperatures ranging from 1200 to 1600 °C under flowing Ar (heating rate, 5 °C/min). Spinning of the PTMB was carried out using the hand-drawing technique in a glove-box filled with dry N_2 [1].

Boron content was measured by a chemical titration method. Element contents of N, H and C were checked by Leco TCH-600 N/H/O and Leco CS-600 C/S analyzers. Fourier transform infrared (FTIR) spectra were taken on a Nicolet Avatar 360 spectrophotometer as KBr pellets. The morphology of the fiber was observed using a scanning electron microscope (JSM-6300, JEOL). X-ray diffraction (XRD) patterns were obtained using Siemens D-5005, Cu Kα radiation. HRTEM images were obtained using a Philips CM-200 microscope operated at 200 kV. Raman spectroscopy was carried out using a RM 2000 spectrometer with an argon ion laser at an excitation wavelength of 632.8 nm. The XPS spectra were obtained with a VG ESCALAB MKΠ instrument (Al Kα excitation). TGA was carried out in flowing air at a heating rate of 10 °C/min (Netzsch STA 449C).

3. Results and discussion

3.1. Characterization and melt-processability of PTMB

Fig. 1 displays the FTIR spectrum of PTMB. The very intense absorption at 1380 cm^{-1} and weak absorption at 728 cm^{-1} can be assigned to the ν(B–N) and δ(B–N), respectively [25,26]. Moreover, strong stretching mode of N–H group at 3436 cm^{-1}, weak NH_2 bending at 1612 cm^{-1} and C–N groups at 1091 cm^{-1}, typical bands for C–H bonds in the range of 2930–3090 cm^{-1} were also noticed. Table 1 illustrates an overview of the most important FTIR absorptions of PTMB and the sample pyrolyzed at 1600 °C.

Polymer fiber was easily prepared by hand-drawing PTMB in the glove box filled with Ar, as described in the literature

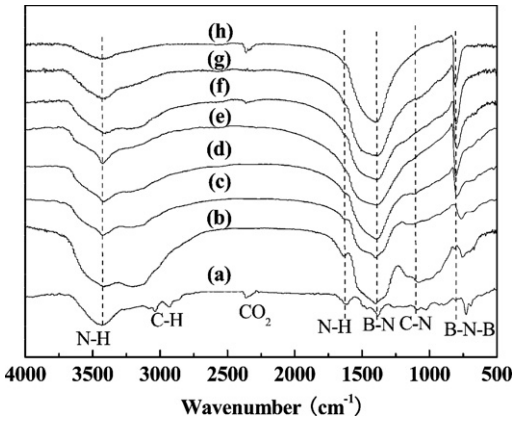


Fig. 1. FTIR spectra of PTMB (a) and PTMB derived sample pyrolyzed at: (b) 400 °C, (c) 600 °C, (d) 800 °C, (e) 900 °C, (f) 1200 °C, (g) 1400 °C and (h) 1600 °C.

Table 1
FTIR absorptions of PTMB and the sample pyrolyzed at 1600 °C.

FTIR bands	Polymer, cm^{-1}	Pyrolyzed sample, cm^{-1}
ν(N–H)	3436/1026	
δ(N–H)	1612	
ν(C–H)	3081/3037/2929/1493	
ν(B–N)	1380	1399
ν(C–N)	1091	
δ(B–N)	728	810

[27]. The fiber in a diameter of about 20 μm exhibited a smooth surface without obvious flaws, as shown in Fig. 2. But even now, just like polycarbosilane fiber [28], the polymer fiber in this study was also very fragile and cannot survive any mechanical shock.

3.2. Conversion from polymer to ceramics

As reported earlier [27], curing in NH_3 could significantly improve the ceramic yield of the polymeric precursor. Here, the pyrolysis behavior of PTMB in NH_3 after curing or not was also examined. Fig. 3 gives the TGA curves of both samples, from which it can be seen that the ceramic yield was different. For the sample without curing, the ceramic yield was 24.07 wt% at 900 °C. While for the sample cured at 80 °C for 10 h, the ceramic yield was enhanced to 38.01 wt%. Moreover, most of the weight loss (98.0 wt% for un-cured and 97.1 wt% for cured, respectively) occurred at 600 °C for both samples. The weight loss between 600 and 900 °C were both less than 3 wt% of the total weight loss at 900 °C.

Prior to curing, the elemental composition (wt%) of PTMB was: B (24.51), C (32.82), N (29.87) and H (10.21), separately, from which the chemical formula was determined to be $BC_{1.20}N_{0.94}H_{4.5}$. It is worthy to note that the carbon content decreased drastically from 32.82 to 24.85 wt% after curing in NH_3 owing to the release of methylamine during curing, similar to that described by Cornu and coworkers [13].

Fig. 4 shows the evolution of carbon content in cured PTMB as a function of temperature under NH_3. As seen, with

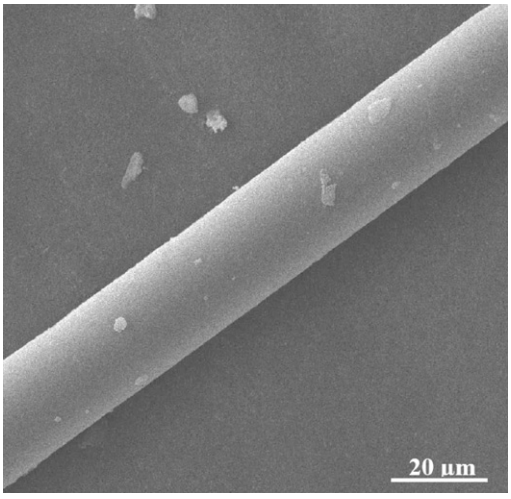


Fig. 2. The typical morphology of the polymer fiber.

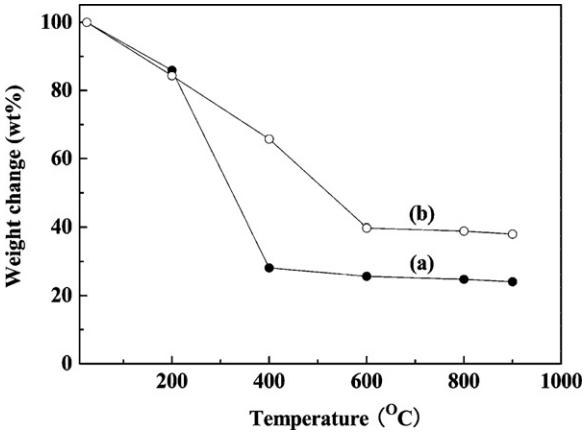
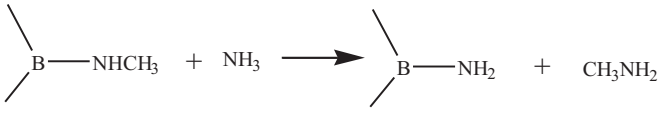


Fig. 3. TGA curves of PTMB in NH_3: (a) un-cured and (b) cured at 80 °C for 10 h.

increasing temperature, the carbon content decreased significantly below 400 °C and occupied about 80.3 wt% of the total carbon removal. Moreover, 93 wt% of carbon was removed at 600 °C. When the temperature was elevated to 800 °C, the carbon content reduced much slower and changed from 5.26 to 0.73 wt%. Above 800 °C, there was no obvious reduction in the carbon content and it reached about 0.37 wt% at 900 °C. However, the carbon content in the N_2 pyrolyzed sample was 5.67 wt% at 900 °C. The difference between two residues was also reflected in the sample color that the former was gray white while the latter was black [29]. The smooth carbon removal in NH_3 was mainly due to the evolution of methylamine caused by transamination reaction (Eq. (1)) and the carbothermal reaction of carbon with NH_3 (Eq. (2)), as shown below [13,30].



(1)

$$C + NH_3 \rightarrow HCN + H_2 \quad (2)$$

The evolution of the structure during pyrolysis was analyzed by FTIR spectra, as represented in Fig. 1. With the temperature increasing, the intensity of C–H, N–H and C–N bands decreased gradually. The C–H band was almost absent in the sample obtained at 400 °C as a result of elimination of methylamine according to Eq. (1) and the intensity of B–N band increased obviously. Also, the intensity of the C–N band at 1026 cm^{-1} showed a reduction supplying the decrease of carbon content. The band attributed to N–H group around 1612 cm^{-1} became weak. Moreover, a wide N–H band corresponding to NH_2 was also observed at about 3209 cm^{-1} caused by transamination reaction, as Eq. (1) displayed.

At 600 °C, the N–H band at 3209 cm^{-1} was not observed and the intensity of C–N band became very weak. Upon further heating up to 900 °C, the C–N band was almost absent, indicating that the carbon content was very low. The result was in consonance with that of EA. Moreover, the FTIR spectrum at 900 °C showed characteristic of BN and those above 900 °C changed little, suggesting that the conversion of polymer to ceramics was almost completed at 900 °C. As the temperature was further increased, the N–H band at around 3436 cm^{-1} became weaker gradually. The sample annealed at 1600 °C showed ν(B–N) and δ(B–N) vibrations at 1399 and 810 cm^{-1}, respectively, confirming the formation of BN [25,26]. Furthermore, the shift of frequency from polymer to ceramics for δ(B–N) was associated with modification of the atomic environment and the crystallization process [21].

Raman spectra of the PTMB pyrolyzed at various temperatures were presented in Fig. 5. Obviously, the diffuse Raman spectra showed characteristic BN near 1500 cm^{-1}. As studied by Rye et al. [20], the large width of the band was characteristic of the amorphous BN. The humps shifted to lower wavenumber due to the increase of crystallinity. It is worth noting that a broad diffuse band at around 2900 cm^{-1} was also present, which was caused by the characteristic of BN overtone. Moreover, there was no emergence of characteristic peak of carbon located around 1600 cm^{-1} [31], indicating complete removal of carbon. Hence, the result of Raman spectra confirmed that of EA above.

The microstructure evolutions of the BN phases were also confirmed by XRD, as displayed in Fig. 6. From Fig. 6, characteristics of amorphous BN can be observed in the residue acquired at 800 °C, similar to that obtained from 900 to 1400 °C. The same shape in XRD patterns indicated very slow crystallization below 1400 °C.

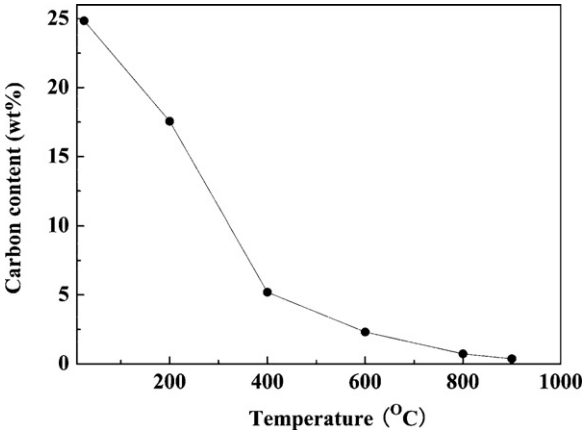


Fig. 4. Evolution of carbon content as a function of temperature.

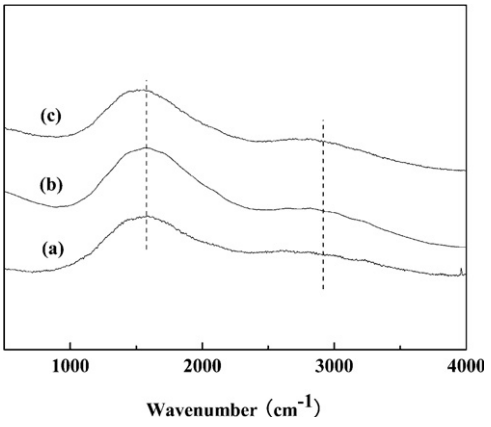


Fig. 5. Raman spectra of PTMB pyrolyzed at various temperatures: (a) 1200, (b) 1400 and (c) 1600 °C.

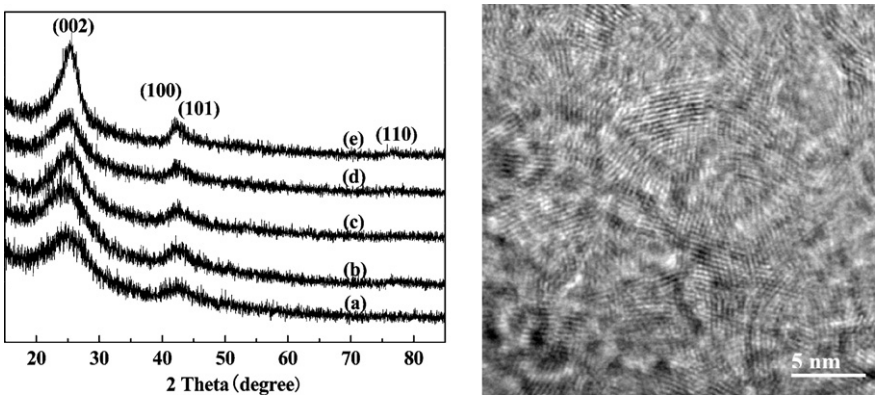


Fig. 6. XRD patterns of the samples obtained at different temperatures: (a) 800 °C, (b) 900 °C, (c) 1200 °C, (d) 1400 °C, (e) 1600 °C and HRTEM image of the sample pyrolyzed at 1600 °C (f).

After heat treated at 1600 °C, the XRD patterns of the sample changed a lot, implying a great phase development happened between 1400 and 1600 °C. Clearly, the full width half maximum (FWHM) of the (0 0 2) plane was narrower compared with that obtained at 800 °C. The sample exhibited no resolution of the (1 0 0) and (1 0 1) plane, showing characteristics of *t*-BN [4,7,22]. Furthermore, a weak peak around $2\theta = 76^{\circ}$ was attributed to the (1 1 0) reflection. However, the value of average interlayer spacing of the (0 0 2) plane d_{002} (0.347 nm) was much higher than that of hexagonal BN (*h*-BN) (0.333 nm) [32], illustrating a lower degree of crystallinity. A higher crystallinity could be acquired by enhancing the pyrolysis temperature [7]. Due to its randomness and partially disordered properties, *t*-BN does not have an accurate crystal structure, and its lattice constants are usually determined by reference to *h*-BN [33]. The lattice constants of the obtained *t*-BN were $a = 2.469$ Å and $c = 6.975$ Å, corresponding to the previous research [33,34]. Additionally, a further investigation of the microstructure for the sample revealed partially ordered BN by HRTEM (Fig. 6(f)) [35], in good agreement with the XRD result.

The composition information of the sample pyrolyzed at 1600 °C was investigated by the XPS spectra, as illustrated in Fig. 7. The survey spectrum (Fig. 7(a)) revealed the presence of B and N elements. Small quantity of C and O were also detected probably as a consequence of the absorption of CO_2 and O_2 on the surface of the sample. The split B1s and N1s spectra were shown in Fig. 7(b) and (c), respectively. The B1s peak at 190.5 eV and the N1s peak at 398.0 eV indicated BN [36]. Carbon element was not detected, pointing out a complete removal of carbon impurity during pyrolysis, in consistent with the EA result. Moreover, the N/B atomic ratio

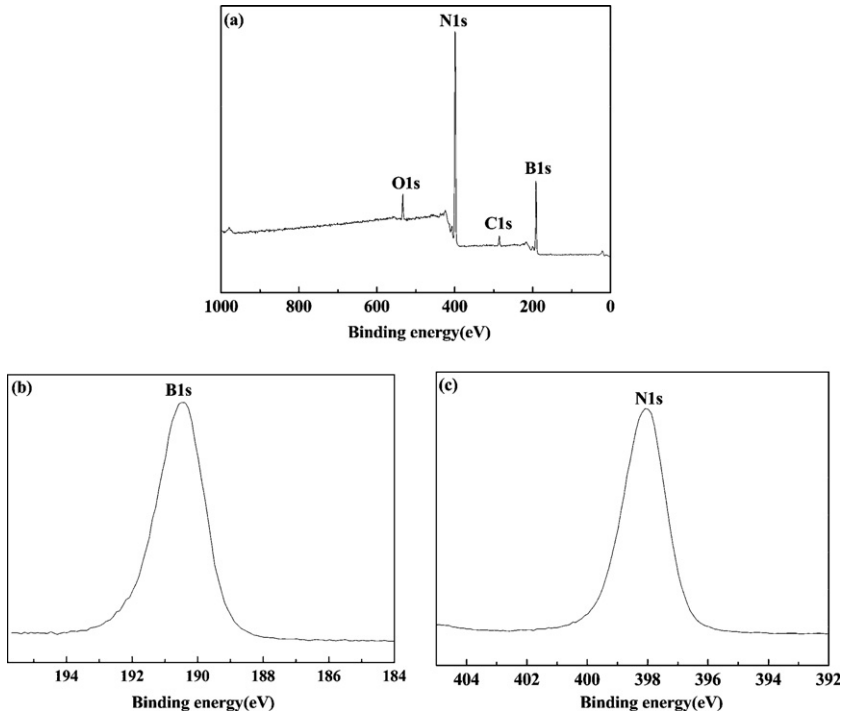


Fig. 7. XPS spectra of the sample obtained at 1600 °C: (a) survey spectra, (b) B1s peak and (c) N1s peak.

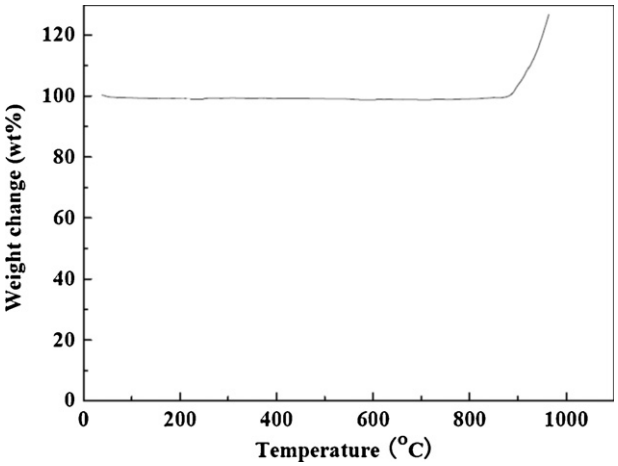


Fig. 8. TGA curve of as-obtained BN heated 10 °C/min in air.

determined by XPS was about 1.00, which was near-stoichiometric, making it possible to possess good oxidation resistance [7].

3.3. Oxidation resistance

The high temperature stability of advanced ceramics is important in determining the suitability for their applications, especially used in oxygen-containing environments [7,35,37,38]. Fig. 8 gives the TGA curve of as-prepared BN in air. It can be seen that the sample displayed less than 3.0 wt% weight change up to 900 °C. Clearly, the sample began to gain weight at about 900 °C due to oxidation of BN. In order to better understand the oxidation behavior of as-obtained BN, the isothermal oxidation process of the BN at 950 °C was also researched, as shown in Fig. 9. As seen, the sample exhibited a continual weight gain till about 80 min.

It is known that BN was oxidized to B_2O_3 when exposed to oxygen/moisture at temperature between 800 and 900 °C, as Eqs. (3) and (4) displayed [39]. The stability to oxidation of BN depends closely on its crystallinity [7,40]. Namely, the BN possessing higher crystallinity exhibits better anti-oxidation property [41,42]. In fact, Cofer and coworkers [41] have tested

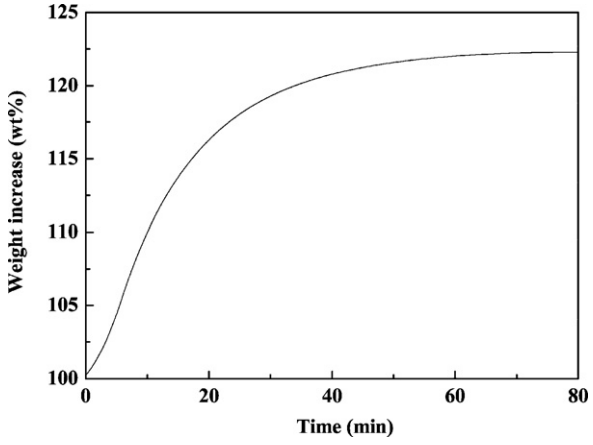


Fig. 9. Isothermal weight gain in air at 950 °C for as-obtained BN (10 °C/min to 950 °C then held).

the oxidation resistance of BN with different crystallinity. They found that BN with d_{002} of 0.333 nm showed a slower weight gain than those with bigger d_{002}. Hence, it is possible to improve the oxidation resistance of as-prepared BN by applying higher heat-treated temperature to elevate the crystallinity.

$$4BN(s) + 3O_2(g) \rightarrow 2B_2O_3(l) + 2N_2(g) \quad (3)$$

$$2BN(s) + 3H_2O(g) \rightarrow B_2O_3(l) + 3H_2(g) + N_2(g) \quad (4)$$

4. Conclusions

In conclusion, PTMB precursor for BN showed good processability by melt-spinning it into fiber. The ceramic yield was greatly improved by curing in NH_3. Pyrolysis under NH_3 led to a complete carbon removal while not under N_2. Moreover, the structure and composition of pyrolysis intermediates were analyzed by FTIR, Raman and EA, illustrating complete conversion of polymer into ceramics at 900 °C. Furthermore, the sample heat treated at 1600 °C showed characteristics of *t*-BN and a near-stoichiometric composition. In addition, the BN displayed good stability in air even up to 900 °C. Higher oxidation resistance would be obtained by applying higher pyrolysis temperature.

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