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Size-selected boron nitride nanosheets as oxygen-atom corrosion resistant fillers

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Experimental

1.8 g PVA (polyvinylalcohol, Beijing Yili Fine Chemicals Co., Ltd., M_w=77000±2200 g/mol) was dissolved in 60 mL water (~90 ^{o}C) and magnetically stirred for 8 h to form homogeneous solution with a PVA concentration of 30 mg/mL. Then 0.6 g crystal BN powder (Alfa Aesar, ≤300 mesh) were added into the PVA solution and sonicated for 8 h in a tip sonic bath (50% of 650W, 3s on, 3s off) with ice cooling. The sonicated dispersions were further centrifuged at 3000rpm (×2304) for 45 min. The top 90% supernatant was collected for further characterization and use. The BNNSs concentration was estimated as ~0.22 mg/mL by TGA analysis of the PVA/BNNSs powder, as the TGA curve shown in Figure S1. The resultant dispersions were expected to contain small BNNSs. These dispersions were then mixed with PVA solution (30 mg/mL) to give a range of composite dispersions with different mass fraction of BNNSs.

After the centrifugation at 3000rpm (×2304g), the sediment was expected to contain medium BNNSs and unexfoliated thick BN flakes. So 40 mL PVA solution (30 mg/mL) was added to dissolve the sediment, followed by sonication for another 2 h by a mild bath sonicator (100 W, 40 KHz) with water cooling. Then 1500rpm (×576g) was used to centrifuge this dispersion and the supernatant was collected. By the method mentioned above, the BNNSs concentration of this dispersion was estimated as ~0.94 mg/mL by TGA analysis of the PVA/BNNSs powder, as the TGA curve shown in Figure S1. The resultant dispersions were expected to contain medium

BNNSs. These dispersions were also then mixed with PVA solution (30 mg/mL) to give a range of composite dispersions with different mass fraction of BNNSs.

After the centrifugation at 1500rpm (×576g), the sediment was expected to contain large BNNSs and unexfoliated thick BN flakes. So 30 mL PVA solution (30 mg/mL) was added to dissolve the sediment, followed by sonication for another 1 h by a mild bath sonicator (100 W, 40 KHz) with water cooling. Then 500rpm (×64g) was used to centrifuge this dispersion and the supernatant was collected. By the method mentioned above, the BNNSs concentration of this dispersion was estimated as ~2.8 mg/mL by TGA analysis of the PVA/BNNSs powder, as the TGA curve shown in Figure S1. These dispersions were also then mixed with PVA solution (30 mg/mL) to give a range of composite dispersions with different mass fraction of BNNSs.

The above composite dispersions were further formed into films for oxygen-atom corrosion test. To form films, after sonication for 20 min, these dispersions were drop cast into high density polyethylene trays and left for 24 h in a vacuum over at 60^{o}C with a pressure of 0.9 bar. Composite films were peeled off and cut into 10×10 mm^2 samples using a die cutter. These films are measured as ~110 μm by micrometer (micrometer screw gauge).

Oxygen-atom exposure experiments were carried out in low earth orbit (LEO) environment ground-based atomic oxygen effect simulation facility in Beijing University of Aeronautics and Astronautics,[1] under a condition of pressure of 0.15 Pa, filament discharge voltage of 120 V, and filament discharge current of 140 mA. The oxygen atom is generated by a plasma-type ground-based atomic oxygen effects simulation facility designed by ourselves (*J. Phys. D: Appl. Phys. 2001,34,2308; Acta Aeronautica Et Astronautica Sinica 2000,21,425*). This facility is with the filament discharge and bound of magnetic field. The temperature in the test is low, less than 50 ^{o}C. The mass loss of Kapton in the atomic oxygen exposure experiments was used as a criterion to calculate the atomic oxygen flux. The calculation formula is $Ft=\Delta M/(\rho A E_y)$ in which F is the effective flow rate of atomic oxygen onto the sample surface and ΔM, ρ, A, t, E_y are mass loss, density, surface area, exposure time, and erosion yield, respectively. Ft is the accumulative atomic oxygen flux exerting on the sample surface. For Kapton, E_y is equal to ~3.0×10^{-24}cm^3/atom. In this study, the accumulative atomic oxygen flux was about 4.7×10^{20} atoms/cm^2, which was approximately equivalent to the accumulative atomic oxygen flux of ~140 days at an orbit of 300 km in LEO.[2] For each mass fraction of PVA/BNNSs composite films, at least three samples were tested and the mass loss averaged.

Scanning electron microscopy (SEM) images were collected by a LEO 1530VP. Atomic force microscopy (AFM) images were captured with a Multimode 8 AFM (Bruker) in ScanAsyst Air mode. Thermogravimetric (TGA) analysis was performed on a TGA/SDTA851 (Mettler Toledo, USA) at 10^{o}C/min in a nitrogen atmosphere. The mass of samples before and after oxygen-atom exposure was measured using a DT-100 balance with a sensitivity of 0.05 mg. X-ray photoelectron spectroscopy (XPS) was obtained by an ESCALAB-250 spectrometer.

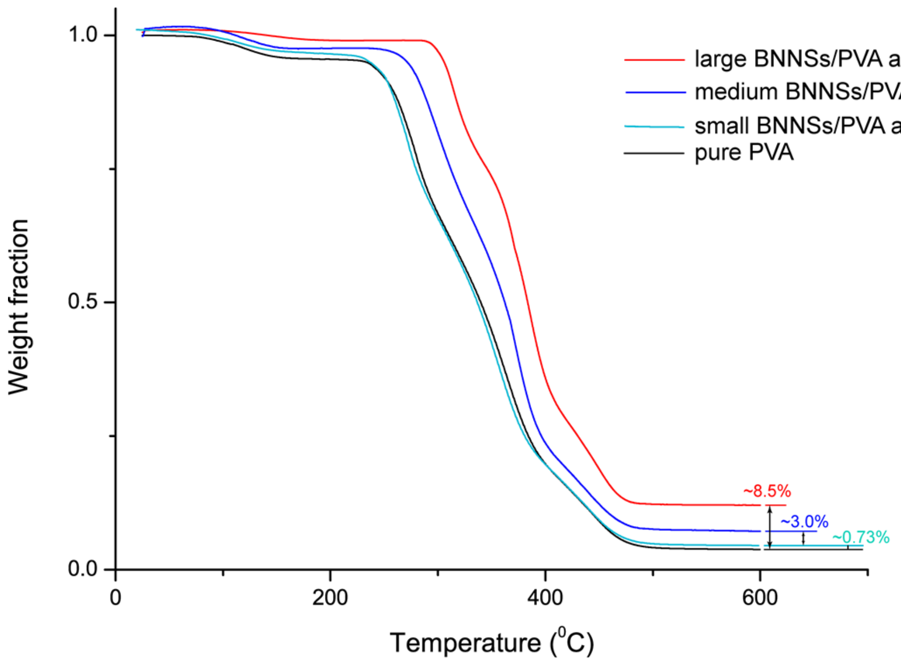

Fig. S1 TGA curves pure PVA and PVA/BNNSs composite powder. The TGA curves of the composites are shifted toward a higher temperature when compared to that of pure PVA, showing the enhancement in thermal stability.

References

1. Yi, M.; Zhang, W.; Shen, Z.; Zhang, X.; Zhao, X.; Zheng, Y.; Ma, S., Controllable functionalization and wettability transition of graphene-based films by an atomic oxygen strategy. J. Nanopart. Res. 2013, 15, 1811.
2. Gouzman, I.; Girshevitz, O.; Grossman, E.; Eliaz, N.; Sukenik, C. N., Thin Film Oxide Barrier Layers: Protection of Kapton from Space Environment by Liquid Phase Deposition of Titanium Oxide. ACS Appl. Mater. Interfaces 2010, 2 (7), 1835-1843.