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1	Highly Stable Tribological Performance and
2	Hydrophobicity of Porous Polyimide Material filled
3	with lubricants in simulated Space Environment
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19 Abstract

20 Space exploitation and development need high-performance materials for spacecraft so as to 21 maintain the long service life and reliability of mechanical equipment. The purpose of the 22 present study was to exploit a new material with durable life, stable friction coefficient and low 23 wear rate in harsh space environments. Two kind of solid-liquid synergetic lubricating 24 composites have been prepared by perfluoropolyethers (PFPE) or chlorinated-phenyl and methyl 25 terminated silicone oil (CPSO) filled in porous polyimide (PPI). Tribological performance and 26 hydrophobicity of oil-filled PPI were evaluated by contact angle analyses and a ball-on-disk 27 tribometer before and after proton irradiation in simulated space environment. After proton 28 irradiation, two composites can maintain stable hydrophobic performance. More importantly, the 29 friction coefficients of CPSO/PPI and PFPE/PPI increased slightly from 0.07 and 0.05 to 0.1 and 30 0.14, respectively. The wear rates of CPSO/PPI and PFPE/PPI also increased slightly from 5.13 \times 10⁻⁵ mm³/Nm and 4.23 \times 10⁻⁵ mm³/Nm to 5.75 \times 10⁻⁵ mm³/Nm and 6.19 \times 10⁻⁵ mm³/Nm, 31 32 respectively. The CPSO/PPI composite showed the smallest change in hydrophobicity, friction 33 coefficient and wear rate before and after proton irradiation. The mechanism of highly stable 34 hydrophobicity and tribological performance was mainly based on continous self-healing suface, 35 stored oil in the pores of PPI can creep to the surface of material to repair the damage induced by 36 Pr irradiation, which ensured that the material has the stable and durable hydrophobicity and 37 tribological properties in proton irradiation environment.

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KEYWORDS: Porous polyimide filled with lubricants; Solid–liquid synergetic lubricating;
Simulated space irradiation; Hydrophobicity and tribological performance.

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42 Introduction

43 The durability of material in rigorous environments is important for the service life and 44 reliability of mechanical equipment, especially for the assemblies applied to space exploitation and development.¹⁻⁵ Spacecraft carries its missions affected by many complicated and 45 46 changeable environmental factors, including thermal cycles ranging hundreds centigrade, ultra-47 high vacuum, high-energy cosmic radiation particles, etc. Among them, protons, electrons, 48 atomic oxygen and ultraviolet rays could severely erode the materials used in the spacecraft and then induce the equipment failure, especially for proton radiation, which caused the worst 49 damage to polymer materials.⁶⁻¹¹ Therefore, developing durable materials that can apply to 50 51 extremely rigorous space environment is still a highly challenging task.

52 In recently years, considerable efforts have been dedicated to develop new materials for space applications, and mostly is studies on the single stable performance by mixing material, 53 54 composite film, etc. The incorporation of carbon nanotubes into the polyimide matrix resulted in the enhanced thermo-optical properties in space environment.⁵ Polyhedral oligomeric 55 56 silsesquioxane incorporated into a Kapton-like polyimide, showing the favorable radiation resistance.^{12, 13} The Pb/PbS composite film could keep stable low frition coefficient in vacuum 57 condition for a long time. ¹⁴ Perfluropolyether-filled anodic aluminum oxide showed highly 58 durable hydrophobicity in simulated space environment.¹⁵ However, these materials could not 59 60 completely suffice the properties requirement of spacecraft special materials.

A desired goal is that the materials own the durable life, stable friction coefficient, low wear rate and durable hydrophobicity in harsh space environments.¹⁶⁻²⁰ In this context, the development of simple and efficient strategy by solid–liquid synergetic lubricating, which would achieve this goal, would be a highly desirable and attractive alternative. Liquid lubricants coated

on the surface of materials would be degraded in space irradiation environment,²¹⁻²³ which was not necessarily the best choice. Herein, we designed a kind of material with porous structures that can be used as a reservoir to store liquid lubricants. When the outermost oil molecules of porous material are degraded by space irradiations, stored lubricants in the porous gaps of material can creep and spread all over the surface of material to maintain the self-healing lubrication, which provides a new strategy to construct new type of materials for the applications of space science.

Polyimide (PI), a kind of important self-lubricating material, widely applied in space science 72 because PI exhibits superior friction and wear characteristics in vacuum.²⁴⁻²⁷ In our previous 73 74 work, PI material has initial high frition coefficient and surface carbonization under proton irradiation environment, which could affect the stability of moving parts.²⁸ A kind of porous PI 75 76 film with an ordered surface was also prepared, but PI film could not apply to lubricating material because of poor mechanical strength.²⁹ On the basis of these work, a new PI composite 77 78 material was designed by filling liquid lubricants into the porous PI material to address the 79 durable lubricating problems in irradiation environment. To the best of our knowledge, there has 80 been no attempt via porous PI filled with lubricants to studied on the tribological behavior and 81 hydrophobicity in extremely rigorous space environment, thereby making an important research 82 work.

In this paper, the porous polyimide (PPI) block material was prepared by cold press and sintering technology using polyimide powder, and then the PPI block was immersed in the liquid lubricants to obtain the oil–containing PPI material. The perfluoropolyethers (PFPE) and chlorinated-phenyl and methyl terminated silicone oil (CPSO) were selected as liquid lubricants owing to excellent characteristics including very low volatility, high thermal resistance, non-

flammability and low surface energy.^{30, 31} Then the PFPE and CPSO–filled PPI materials were subjected to proton irradiation test in simulated space environment because proton irradiation seriously affected tribological behavior of the PI materials.^{21, 32} The hydrophobicity and tribological properties of oils–filled PPI materials were evaluated by contact angle (CA) analyses and a ball–on–disk tribometer. Multiple characterization techniques were employed to investigate changes in surface structures using attenuated total reflectance infrared spectroscopy (ATR-FTIR), micro–Raman spectroscopy and scanning electron microscope (SEM).

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96 Experimental

97 Materials

98 The used PI powders with the particle size of 400 mesh were purchased from Shanghai Research Institute of Synthetic Resins, which has the density of 1.35 g/cm³ and the glass 99 100 transition temperature 250 °C. The PPI material was prepared by cold press and sintering 101 technology, during which the PI powders were cold pressed in mold under a pressure of 30 MPa for 30 min and then sintered in an oven at 300 °C for 240 min to form a $20 \times 12 \times 15$ mm³ block 102 which was finally cut into $20 \times 12 \times 2 \text{ mm}^3$ blocks for irradiation and wear test. The SEM picture 103 104 of cross section of PPI sample was shown in Fig. 1. The prepared PPI samples were firstly dried 105 in the vacuum under 120 °C for 2 h and then quickly immersed in the selected lubricating oil for 106 24 h in the vacuum under 120 °C to fully infiltrate into the inner pore, followed by wiping the oil 107 in the surface with cotton cloth. The liquid lubricants used were chlorinated-phenyl and methyl 108 terminated silicone oil (CPSO) and perfluoropolyethers (PFPE, Fomblin M30), and the 109 corresponding chemical formulation were shown in Fig. 2. CPSO was supplied by Lanzhou 110 Institute of Chemical Physics. PFPE was obtained from Solvay solexis. Inc. and used as received.



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protons was determined to be about 6.25×10^{15} ions/cm²·s. The exposure time of Pr irradiation was controlled as 5 min which was selected due to Pr irradiation possessing higher energy for corroding organic compounds molecules.^{15, 21}

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 $\begin{bmatrix} \mathbf{GCr \, 15 \, ball} \\ \mathbf{r} \\ \mathbf{b} \\ \mathbf{PPI} \end{bmatrix} \begin{bmatrix} (1) \, V = \left[\frac{\pi r^2}{180} \arcsin(\frac{b}{2r}) - \frac{b}{2} \sqrt{r^2 - \frac{b^2}{4}} \right] \pi d \\ (2) \, K = V/PL \\ \mathbf{PPI} \end{bmatrix}$

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Fig. 4 Calculation formula of the wear rate.

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132 The friction and wear behavior of the bare PPI and oil-filled PPIs before and after Pr 133 irradiation against GCr15 steel ball were tested on a ball-on-disk tribometer in a vacuum level of 3×10^{-4} Pa. The GCr15 steel ball with a standard 3 mm diameter have the chemical composition 134 (in wt%): Mn (0.20-0.40), Si (0.15-0.35), Cr (1.30-1.65), C (0.75-0.85), P (≤0.026), S (≤0.020) 135 136 and Fe balance. The hardness and elastic modulus are 6.9 GPa and 208 GPa, respectively. The 137 steel ball slid on sample disk that rotate at a speed of 0.126 m/s under the load of 5 N for 1800 s 138 with a rotational diameter of 10 mm. Fig. 4 shows the calculation for wear rate. Where b and d 139 respectively denote the width and the diameter of the wear track (10 mm), r refers to the radius of the steel ball, V is the wear volume loss of PPI block (mm³), K (mm³·Nm⁻¹) correspond to 140 141 wear rate value, P is the applied (N) load and L is the sliding distance (m). In order to minimize the error, three specimens were tested under each condition to attain the average wear rate of 142 143 samples.

144 Characterization

145 Contact angles (CA) were measured using a DSA-100 optical contact-angle meter (Kruss,

Germany) at room temperature (20 °C) by injecting 5 µL double distilled water on the samples 146 147 surface. Images were captured with a Digital Camera (Sony, Japan) and the average CA values 148 were obtained by testing the same sample at five different positions. The infrared spectroscopic 149 measurements were carried out on a Nexus 870 FTIR spectrometer (Nicolet, USA) using the 150 attenuated total reflection (ATR) technique with a germanium crystal. A JEM-5600LV scanning 151 electron microscope (SEM, JEOL, Japan) and an optical microscope were used to observe 152 surface micrographs of samples. The ingredients of worn surfaces on steel ball were analyzed by 153 a laser micro-Raman spectroscopy (LabRam HR800, Japan) at an excitation wavelength of 633 154 nm and energy dispersive X-ray spectroscopy (EDS).

155

156 **Results and discussion**

157 Hydrophobicity





Fig. 5 Contac tangles of bare PPI and oil-filled PPI before and after Pr irradiation.

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The CA of water was measured to investigate the hydrophobicity performance of bare PPI and oil-filled PPI before and after Pr irradiation, and the results were presented in Fig. 5. Bare PPI, without any liquid lubricant, had a water CA of 90 °. After impregnated with CPSO and PFPE liquid lubricatin oil, the CA increased from 90 ° to 113 ° and 109 °, respectively. After Pr irradiation for 5 min, it can be easily seen that the CA of bare PPI, CPSO/PPI and PFPE/PPI

- increased to 112°, 116° and 120°, respectively. The above results indicated that filling the oils
 in PPI can enhance its hydrophobic property. In addition, these new composite can maintain
 stable hydrophobic performance after Pr irradiation compared with the bare PPI, especial for the
 CPSO/PPI sample.
- 170 Tribological performance
- 171



Fig. 6 The friction coefficient variation (a) and the wear rate (b) of bare PPI and oil-filled PPI before and after
 Pr irradiation.

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Friction coefficient and wear rate are the main indexs to evaluate tribological property. The 176 177 changes in friction and wear behaviors of PPI and oil-filled PPI against GCrl5 steel balls before 178 and after Pr irradiation were comparatively investigated using a ball-on-disk tribometer. Fig. 6a 179 display the friction coefficient variations of bare PPI, CPSO/PPI and PFPE/PPI before and after 180 Pr irradiation. Before Pr irradiation, it can be seen that the friction coefficient of the bare PPI 181 sample is the highest with an obvious fluctuation in a range of 0.26-0.29. While, the friction 182 coefficients of CPSO/PPI and PFPE/PPI remained respectively very stable at about 0.07 and 0.05, 183 which is almost 1/4 and 1/6 of the friction coefficient of the bare PPI. After Pr irradiation, the 184 friction coefficient curve of bare PPI sample exhibited a drastic fluctuation, in which the initial 185 friction coefficient increased to 0.34 and the steady friction coefficient decreased to 0.10.

186 Whereas, the friction coefficients of CPSO/PPI and PFPE/PPI increased slightly from to 0.07 and 187 0.05 to 0.10 and 0.14, respectively, and the curve is still stable. The bar charts in Fig. 6b present 188 the wear rates of PPI, CPSO/PPI and PFPE/PPI before and after Pr irradiation. The wear rates of 189 all samples have different degrees of increment after Pr irradiation. As for bare PPI, the wear rate after Pr irradiation is 18.36×10^{-5} mm³/Nm, which is about 2.3 times of that before Pr irradiation 190 $(8.06 \times 10^{-5} \text{ mm}^3/\text{Nm})$. In the case of CPSO/PPI, the wear rate after Pr irradiation is 5.75×10^{-5} 191 mm³/Nm, which is about 1.1 times of that before Pr irradiation (5.13 \times 10⁻⁵ mm³/Nm). As for 192 PFPE/PPI, the wear rate after Pr irradiation is $6.19 \times 10^{-5} \text{ mm}^3/\text{Nm}$, which is about 1.5 times of 193 that before Pr irradiation $(4.23 \times 10^{-5} \text{ mm}^3/\text{Nm})$. Therefore, filling oil in PPI is effective in 194 195 decreasing the friction coefficient and wear rate to achieve super low-friction and wear resistance 196 materials. Meanwhile, the friction and wear behavior of bare PPI is very sensitive to Pr 197 irradiation, but the oil-filled PPI showed wonderful stability under Pr irradiation environment. 198 Especially for CPSO/PPI that exhibited the smallest change in friction and wear behavior before 199 and after Pr irradiation.

200 Microstructural analysis

To investigate the reason of the durable and stable hydrophobicity and tribological properties of the oil-filled PPI under Pr irradiation environment, the chemical structures, surface morphologies, and worn surface of the oil-filled PPI and steel ball were characterized by SEM, ATR-FTIR, Raman and EDS.

The surface morphologies of PPI and oil-filled PPI were observed by SEM before and after Pr irradiation, and the results were shown in Fig. 7. Compared to the bare PPI, the surfaces of CPSO/PPI and PFPE/PPI are relatively smooth, indicating that both CPSO and PFPE completely covered the rough PPI surface. After Pr irradiation, the surface of bare PPI was seriously eroded

- to become much rougher, but the CPSO/PPI and PFPE/PPI still keep relatively smooth surfaces.
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Fig. 7 SEM surface morphologies of PPI and oil-filled PPI before and after Pr irradiations.

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Fig. 8 FTIR-ATR spectra of PPI and oil-filled PPI before and after Pr irradiation.

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cm⁻¹ (CF), 1230 cm⁻¹ (CF₂).^{15, 33} The chemical degradation of material induced by irradiation 223 can be conveniently investigated by following the intensity changes of various FTIR peaks.³⁴ In 224 225 addition, many research results showed that Pr irradiation could induce chain scission reaction 226 occurring in organic polymer materials and then the the intensity of the characteristic peaks may reduce obviously.^{33, 35-37} In this test, after Pr irradiation the intensity of the characteristic peaks 227 228 for PPI in both bare PPI and oil-filled PPI decreased greatly. In contrast, the intensity of the 229 characteristic peaks for both CPSO and PFPE oil reduced very slightly, which is not consistent 230 with evidence in the reference that the Pr irradiation can seriously erod the oil molecules. The 231 reason for this inconsistent is that the stored oil molecules in the prorous structure of PPI tends to spread out from the pore structure to the top surface of PPI due to the low surface energy.³⁸ 232

233 The worn surfaces of steel ball were analyzed in detail by SEM, Raman and EDS, and the 234 results were presented in Fig. 9. As for the bare PPI, there was accumulated continuous transfer 235 layer presented on the steel ball surface before and after Pr irradiation (Fig. 9a). As shown in Fig. 236 9b, the Raman spectra of transfer layer of bare PPI before irradiation did not have specific peaks, 237 which indicated that the composition of transfer layer was considered as polymer-like carbon. On 238 the other hand, the spectrum of transfer layer of bare PPI after irradiation displayed two broad peaks at 1357 cm⁻¹ and 1580 cm⁻¹ that correspond to the D and G bands of disordered and 239 240 ordered graphite, which indicated that a graphite-like structure was formed in the transfer layer. 241 In contrast, there was no continuous transfer layer on the steel ball surfaces of the oil-filled PPI 242 before and after Pr irradiation (Fig. 9a). In addition, no characteric peaks in Raman spectra were 243 observed most likely because the changes occurred on the steel ball was out of the detection limit 244 of the applied Raman system. Moreover, the EDS was used to analyze the elements on the steel 245 ball. As shown in Fig. 9c, elements Si and F were detected in the steel ball surfaces of the oil-

- filled PPI before and after Pr irradiation, indicating that the stell balls surface of the oil-filled PPI
 contained lubircating oil which can prevent the solid-to-solid contact and then reduce the friction
 coefficient and wear rate.
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257 According to the above analysis results, it can be easy to deduce the mechanism of the 258 stability and durability of the hydrophobicity and tribological performance of oil-filled PPI under 259 Pr irradiation, and the schematic was illustrated in Fig. 10. After the Pr irradiation in simulated 260 space environment, the bare PPI has a significant change in hydrophobic properties and 261 tribological performance because Pr irradiation could erode the surface structure. Though the 262 surface of oil-filled PPI was also suffered some damage, the stored oil molecules in the prorous 263 structure of PPI tends to creep on the surface of PPI especilly for the CPSO oil due to the lower 264 surface energy. Therefore, the ability to withstand Pr irradiation damage was mainly based on 265 continous self-healing suface as a result of stored oil in the prorous structure of PPI, and the lubricants oils could spread out from its micropores onto the contact surface to form a 266 267 homogeneous and plain oil film which enhanced hydrophobicity and reduced its friction 268 coefficient and wear rate. Meanwhile, a balance may be maintained between the irradiation 269 damage and self-healing of oil-filled PPI, which ensured that the material has the stable and 270 durable hydrophobicity and tribological properties in Pr irradiation environment.

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277 In sum, two kind of PPI composite materials were designed and fabricated by filling PFPE or 278 CPSO oils into the PPI pore. Oil-filled PPI showed the highly stable and durable hydrophobicity 279 and tribological performance compared with the bare PPI, especially for the CPSO/PPI 280 composite which had the smallest changes in hydrophobicity, friction coefficient and wear rate 281 before and after Pr irradiation. Combining with the characterizations of SEM, ATR-FTIR, Raman 282 and EDS, Pr irradiation could induce the surface damage of all test samples. The graphite-like 283 substance was formed on bare PPI surface, but the CPSO/PPI and PFPE/PPI still keep relatively 284 smooth surfaces. These excellent performances of oil-filled PPI in simulated space environment 285 was mainly attributed to the stored oil in the pores of PPI tends to spread all over the PPI surface 286 to maintain the self-healing hydrophobicity and lubrication.

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- 293 References
- 294 1 L. Rapoport, N. Fleischer and R. Tenne, *Adv. Mater.*, 2003, 15, 651-655.
- 295 2 Y. Zhang, X. Yin, J. Wang and F. Yan, *RSC Adv.*, 2014, 4, 55752-55759.
- J. G. Smith Jr, J. W. Connell, D. M. Delozier, P. T. Lillehei, K. A. Watson, Y. Lin, B. Zhou and Y. P. Sun, *Polymer*, 2004, 45, 825-836.
- 298 4 J. G. Smith, J. W. Connell, K. A. Watson and P. M. Danehy, *Polymer*, 2005, 46, 2276-2284.
- 299 5 N. Atar, E. Grossman, I. Gouzman, A. Bolker and Y. Hanein, *ACS Appl. Mater. Inter.*, 2014, 6, 2040020407.
- 301 6 H. Koshiishi and H. Matsumoto, J. Atmos. Sol.-Terr. Phy., 2013, 99, 129-133.
- 302 7 X.-H. Zhao, Z.-G. Shen, Y.-S. Xing and S.-L. Ma, Polym. Degrad. Stab., 2005, 88, 275-285.
- 303 8 M. Tagawa, M. Muromoto, S. Hachiue, K. Yokota, N. Ohmae, K. Matsumoto and M. Suzuki, *Tribol. Lett.*,
 304 2005, 18, 437-443.
- 305 9 V. A. Shuvalov, N. I. Pis'mennyi, G. S. Kochubei and N. A. Tokmak, *Cosmic Res.*, 2014, 52, 99-105.
- 306 10 M. Tagawa, K. Yokota, K. Kishida, A. Okamoto and T. K. Minton, *ACS Appl. Mater. Inter.*, 2010, 2, 1866307 1871.
- 308 11 P. A. Bertrand, *Tribol. Lett.*, 2010, 40, 187-198.
- 309 12 T. K. Minton, M. E. Wright, S. J. Tomczak, S. A. Marquez, L. Shen, A. L. Brunsvold, R. Cooper, J. Zhang,
- 310 V. Vij, A. J. Guenthner and B. J. Petteys, ACS Appl. Mater. Inter., 2011, 4, 492-502.
- 311 13 G. Song, X. Li, Q. Jiang, J. Mu and Z. Jiang, *RSC Adv.*, 2015, DOI: 10.1039/C4RA14727H.
- 312 14 G. Ma, B. Xu, H. Wang, S. Chen and Z. Xing, ACS Appl. Mater. Inter., 2014, 6, 532-538.
- 313 15 R. Guo, H. Hu, Z. Liu, X. Wang and F. Zhou, *RSC Adv.*, 2014, 4, 28780-28785.
- 314 16 T. K. Minton, J.-F. Roussel and J.-F. Roussel, *ACS Appl. Mater. Inter.*, 2010, 2, 2687-2688.
- 315 17 G. Li, X. Liu and T. Li, Composites Part B-Eng., 2013, 44, 60-66.
- 316 18 K. A. Watson, F. L. Palmieri and J. W. Connell, *Macromolecules*, 2002, 35, 4968-4974.
- 317 19 R. L. Fusaro, *Tribol. Int.*, 1990, 23, 105-122.
- 318 20 M. T. Tagawa, Masahito and K. Yokota, *Acta Astronaut.*, 2008, 62, 203-211.

- 319 21 X. Liu, L. Wang, J. Pu and Q. Xue, Appl. Surf. Sci., 2012, 258, 8289-8297.
- 320 22 X. F. Liu, J. B. Pu, L. P. Wang and Q. J. Xue, J. Mater. Chem. A, 2013, 1, 3797-3809.
- 321 23 X. Liu, L. Wang and Q. Xue, *Tribol. Int.*, 2013, 60, 36-44.
- 322 24 I. Gofman, B. D. Zhang, W. C. Zang, Y. Zhang, G. L. Song, C. H. Chen and Y. Li, J. Polym. Res., 2013, 20,
- 323 258-267.
- 324 25 B. Liu, M. Ji, J. G. Liu, L. Fan and S. Y. Yang, *High Perform. Polym.*, 2013, 25, 907-918.
- 325 26 F. Xiao, K. Wang and M. Zhan, J. Mater. Sci., 2012, 47, 4904-4913.
- 326 27 M. Iwata, A. Ohnishi, H. Hirosawa and F. Tohyama, J. Spacecr. Rockets, 2001, 38, 504-509.
- 327 28 M. Lv, F. Zheng, Q. Wang, T. Wang and Y. Liang, *Wear*, 2014, 316, 30-36.
- 328 29 C. Wang, Q. H. Wang and T. M. Wang, *Langmuir*, 2010, 26, 18357-18361.
- 329 30 L. J. Weng, H. Z. Wang, D. P. Feng, W. M. Liu and Q. J. Xue, Ind. Lubr. Tribol., 2008, 60, 216-221.
- 330 31 M. Marchetti, M. H. Meurisse, P. Vergne, J. Sicre, M. Durand and J. Durand, in *Proceedings of the 8th* 331 *European Space Mechanisms and Tribology Symposium*, ed. D. Danesy, 1998, vol. 438, pp. 233-238.
- 332 32 M. Lv, Y. Wang, Q. Wang, T. Wang and Y. Liang, *Radiat. Phys. Chem.*, 2015, 107, 171-177.
- 333 33 R. Huszank, S. Z. Szilasi and D. Szikra, J. Phys. Chem. C, 2013, 117, 25884-25889.
- 334 34 E. J. Petersen, T. Lam, J. M. Gorham, K. C. Scott, C. J. Long, D. Stanley, R. Sharma, J. Alexander Liddle,
- B. Pellegrin and T. Nguyen, *Carbon*, 2014, 69, 194-205.
- 336 35 R. Huszank, D. Szikra, A. Simon, S. Z. Szilasi and I. P. Nagy, *Langmuir*, 2011, 27, 3842-3848.
- 337 36 M. Porubská, O. Szöllős, A. Kóňová, I. Janigová, M. Jašková, K. Jomová and I. Chodák, *Polym. Degrad.*338 *Stab.*, 2012, 97, 523-531.
- 339 37 B. X. Liu, X. Q. Pei, Q. H. Wang, X. J. Sun and T. M. Wang, *Appl. Surf. Sci.*, 2011, 258, 1097-1102.
- 340 38 P. Saravanan, N. Satyanarayana, D. H. Minh and S. K. Sinha, *Wear*, 2013, 307, 182-189.