

Fluorinated Polyurethane-Based Enameled Wires with a Low Friction Coefficient

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Cite This: *ACS Omega* 2021, 6, 4719–4725



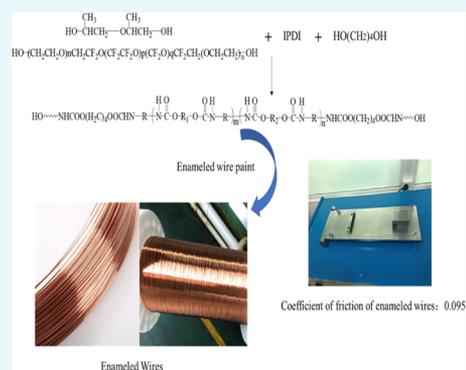
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ABSTRACT: Fluorinated polyurethane (FPU) with a different fluorine content was prepared using perfluoropolyether glycols, poly(propylene glycol), and isophorone diisocyanate as starting materials, and 1,4-butanediol as a chain extender. The structure and molecular weight of FPU were characterized by Fourier transform infrared spectroscopy and gel permeation chromatography. A solution of FPU in xylene and cresol was then coated on copper wires using an enameled machine to prepare enameled wires. The friction coefficient and adhesion performance of the enameled wires were tested. The friction coefficient of the as-prepared enameled wires reached 0.095, which was much lower than 0.149 of the polyurethane without fluorine. FPU-based enameled wires also showed good mechanical performances and increased breakdown voltages. In addition, FPU exhibited good hydrophobic and oleophobic characterization.



1. INTRODUCTION

Enameled wires are widely used in precision coils, relays, home appliances, telecommunications, and meters.^{1–3} There are mechanical stresses acting on the enameled wire during the coil winding and embedding process, as well as the operation of electrical products as components, which may lead to elongation of the enameled wire and wear of the paint film. In particular, the current coil winding technology uses high-speed automatic winding machines.^{4,5} The enameled wire is subjected to friction and impact in the winding process. The insulation layer of the enameled wire is vulnerable to mechanical damage, which causes the decreased insulation performance. In addition, due to poor lubrication, the enameled wire with a small diameter will be thinned, and the cross-sectional area of the enameled wire will become smaller, which might cause the enameled wire to break and the coil to scrap.

To reduce the friction coefficient, the enameled wire is often coated with paraffin or other lubricants.⁶ However, too much paraffin wax mainly affects the combination effect of an injection molding resin and an enameled coil during the follow-up processing of the coil. In addition, the paraffin wax coating also affects the electrical characteristics of parts of electronic components such as the contact effect at the contact point. Therefore, the development of low friction coefficient ultrafine enameled wire products without coating with paraffin lubricants is an urgent demand for the enameled wire industry.

Another route to reducing the friction coefficient of enameled wires is to introduce a low friction factor lubricant

molecule containing a specific functional group into a matrix resin molecular chain through polymerization or other reactions, thereby imparting low friction properties or self-lubricating properties to the polymer and serving as a resin matrix for the enameled wire paint.⁷ Ressel et al.⁸ synthesized polyimide and poly(dimethylsiloxane) block copolymers and cured them into enameled wire paint. Depending on the poly(dimethylsiloxane) of a block copolymer, the friction coefficient was reduced. However, the decrease in the friction coefficient for the enameled wires is limited due to the own characteristics of organic siloxane. It is important to prepare polymers with a low friction coefficient by introducing other groups. Fluorine-containing polymers have low intermolecular cohesion, low surface free energy, and low friction coefficient.^{9–11} Zhou et al.¹² reported that polyimide/fluorinated graphene nanocomposite films had a lower friction coefficient of 0.3 due to the existence of fluorine. Zhao et al.¹³ prepared waterborne polyurethane/fluoride polyphosphazene microsphere composites with enhanced thermal stability and friction properties, and the minimum friction coefficient was 1.07. Zhou et al.¹⁴ found that the introduction of fluorosiloxane copolymers into polyurethane acrylate coating

Received: November 11, 2020

Accepted: February 3, 2021

Published: February 11, 2021



could enhance the abrasion resistance of the hybrid film. Therefore, it is expected to significantly reduce the friction coefficient by the introduction of fluorine-containing groups in the polymer molecule chain.

Polyurethane (PU) enameled wire is an important variety in enameled wire products due to its direct solderability and hydrolysis resistance.^{15,16} The development of PU-enameled wire with a low friction coefficient is the guarantee of ultrahigh processing speed and the long life of high-end electronic and electrical coils. Although there are many research reports on polyurethane coatings,¹⁷ the friction coefficient of polyurethane wire enamels is still decreased by coating with paraffin wax, which is forbidden for components of high-end electronic and electrical products. Therefore, the introduction of fluorine-containing groups in the PU molecule chain will be a key to develop low friction coefficient PU-enameled wires without coating with paraffin lubricants. Tonelli et al.¹⁸ reported the lowest friction coefficient of 0.28 for fluorine-containing thermoplastic polyurethanes prepared by a three-step polymerization technique using poly(tetramethylene glycol), polybutadienediol, poly(fluoroalkylene) diol, and diisocyanate (MDI) as starting materials and 1,4-butanediol as a chain extender. Li et al.¹⁹ blended thermoplastic polyurethanes with fluorinated ultrahigh-molecular-weight polyethylene to obtain the composite with the friction coefficient of 0.19. The lowered friction coefficient was attributed to the low surface free energy of fluorine-containing groups.^{18,19} In this paper, the fluorine element was introduced into the polyurethane main chain by a simple two-step method using perfluoropolyether polyol, poly(propylene glycol) (PPG), and isophorone diisocyanate (IPDI) as starting materials and 1,4-butanediol as a chain extender. The lowest friction coefficient of 0.095 for fluorinated polyurethane-enameled wires was obtained. Therefore, it is of great significance to the development of high-end electronic devices.

2. RESULTS AND DISCUSSION

FPU100 was prepared using perfluoropolyether glycol and IPDI as starting materials. PU was synthesized using PPG and IPDI as starting materials. Fluorinated polyurethane copolymers with different contents of fluorine (co-FPU20 and co-FPU50) were synthesized using the reaction of perfluoropolyether glycol, PPG, and IPDI by replacing 20 and 50 mol % of PPG with perfluoropolyether glycol.

The infrared (IR) spectra of PU, co-FPU20, co-FPU50, and FPU100 are shown in Figure 1. The absorption peaks of 3325 and 1700 cm^{-1} are attributed to the stretching vibration of $-\text{NH}$ and $-\text{C}=\text{O}$ from the urethane. The bending vibration absorption peak of $-\text{NH}$ in the carbamate is at 1536 cm^{-1} . The peak at 1232 cm^{-1} is due to the $\text{C}-\text{O}$ bond. The peak at 1099 cm^{-1} is assigned to the stretching vibration of $\text{C}-\text{O}-\text{C}$. No peak of $-\text{NCO}$ is observed at 2260–2280 cm^{-1} , indicating that $-\text{NCO}$ has completely reacted. Compared to the IR spectrum of PU, a peak at 1138 cm^{-1} attributed to the stretching vibration of $\text{C}-\text{F}$ is observed for co-FPU20, co-FPU50, and FPU100.

Molecular weights of different samples are shown in Table 1. The molecular weights of PU, co-FPU20, and FPU100 are more than 40 000, while that of co-FPU50 is 20 543. The co-FPU50 was synthesized by replacing 50 mol % of PPG with perfluoropolyether glycol. In this case, the proportion of fluoropolyether glycol is higher, the poor compatibility between fluoropolyether glycol and PPG leads to delamination,

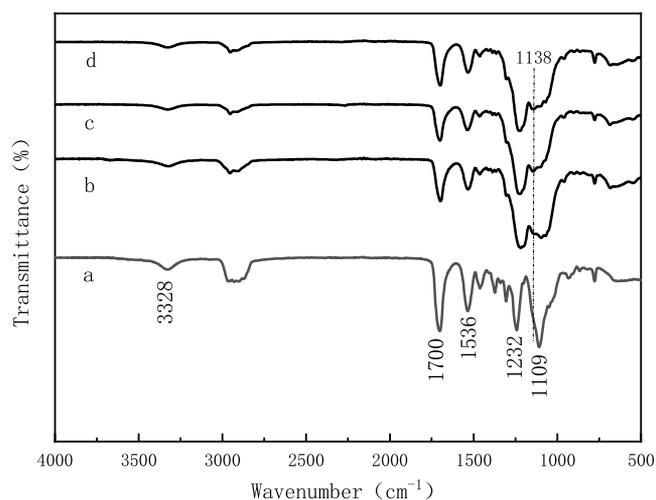


Figure 1. Fourier transform infrared spectroscopy (FTIR) spectra of PU (a), co-FPU20 (b), co-FPU50 (c), and FPU100 (d).

Table 1. Molecular Weight of Different Samples

sample ID	perfluoropolyether glycol content (mol %)	molecular weight (M_n)
PU	0	41 084
co-FPU20	20	42 084
co-FPU50	50	20 543
FPU100	100	45 877

which might affect the process of polymerization. Therefore, the molecular weight of co-FPU50 is lower. However, it is high enough for the coating. Combined with the IR spectra, FPU5 were all successfully synthesized by controlling the ratio of perfluoropolyether glycols to PPG.

X-ray photoelectron spectroscopy (XPS) survey scans of FPU100 film before and after etching are shown in Figure 2 and Table 2. From the spectrum before etching with argon ion, the strong F 1s signal and the weak signals of C 1s, O 1s, and N 1s are observed (Figure 2a). After etching, there is an obvious decrease of F 1s and a large increase of the C 1s signal (Figure 2b). As shown in Table 2, the atomic mass content of the fluorine for the FPU100 film before etching is 28.91%,

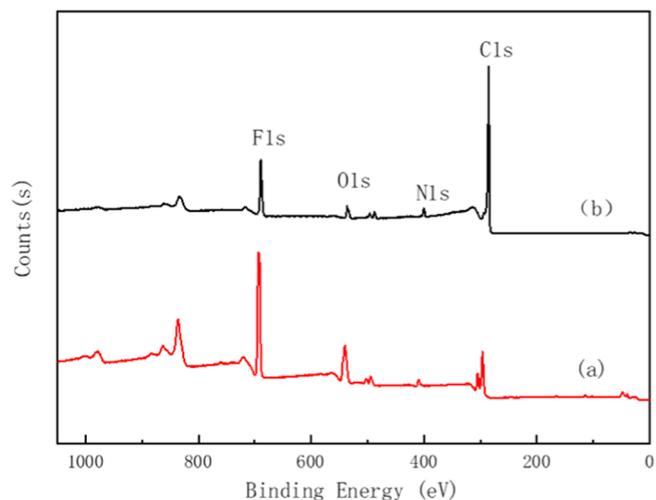


Figure 2. XPS survey scan of the FPU100 film (a) before etching and (b) after etching.

Table 2. Atomic Compositions of FPU100

surface	C/wt %	O/wt %	F/wt %	N/wt %
before etching	47.85	18.42	28.91	4.82
after etching	81.14	4.23	11.06	3.56

which is much higher than that of the FPU100 film after etching (11.06%), indicating the enrichment of the fluoroalkyl segments at the film–air interface.

Table 3 lists the friction coefficient of the PU/co-FPU20/co-FPU50/FPU100-enameled wires. Table 4 lists the comparison

Table 3. Friction Coefficient of Different Enameled Wires

sample ID	perfluoropolyether glycol content (mol %)	coefficient of friction
PU	0	0.149 ± 0.005
co-FPU20	20	0.148 ± 0.007
co-FPU50	50	0.132 ± 0.001
FPU100	100	0.095 ± 0.005

Table 4. Comparison of Friction Coefficients of Different Materials

no.	polymer	friction coefficient	ref
1	FPU-enameled wires	0.095	this work
2	fluorine-containing thermoplastic polyurethanes	0.28	18
3	fluorinated-ultrahigh-molecular-weight polyethylene-modified thermoplastic polyurethanes	0.19	19
4	waterborne polyurethane/fluoride polyphosphazene microsphere composites	0.107	13
5	polyurethane/graphene oxide hybrid wall microcapsules	0.150	20
6	mesoporous-silica-modified polyurethane	1.70	21
7	fluorine-modified polyimide film	0.35	22
8	fluorinated graphene-modified polyimide film	0.3	12

of this work to the reported literature as well as the revised manuscript. The coefficient of friction of PU-enameled wire without the introduction of the fluorine element is 0.149. When the content of perfluoropolyether glycol is 20 mol%, the friction coefficient of co-FPU20 is 0.148. With the further increase in the content of perfluoropolyether glycol, the friction coefficients of co-FPU50 and FPU100 are 0.132 and 0.095, respectively. Compared with PU, the friction coefficient of FPU100-enameled wire is reduced by 36.2%. As shown in Table 4, the friction coefficient of FPU100-enameled wire is also much lower than those of other fluorinated coatings and modified PU.^{20–22} It can be seen that as the content of the fluorine element in the polymer increases, the friction coefficient of the enameled wires decreases. The fluorine atoms with strong electronegativity and larger radius than those of hydrogen atom have a shielding effect on the carbon atoms of the molecular chain so that the interaction force between the molecules is small and the cohesion energy is low. In addition, the fluorine-containing polymer has extremely low surface energy. In the case of a fluorine-containing copolymer, the enrichment of fluorine-containing segments to the surface is thermodynamically and kinetically favored by the low surface energy and high mobility of the fluorine-containing chain.^{23,24} In summary, the enrichment of the fluorine-containing

segments to the surface and the unusually low surface energy are all responsible for the low friction coefficient. Therefore, FPU can reduce the friction coefficient without adding an external lubricant. With the increase of fluorine content in FPU, the groups containing more fluorine are enriched on the surface and the surface free energy is lowered. Therefore, the FPU100-enameled wire with the highest fluorine content in FPU shows the lowest friction coefficient.

The mechanical properties of the PU/co-FPU20/co-FPU50/FPU100-enameled wires are shown in Table 5. The

Table 5. Mechanical Properties of Different Enameled Wires

sample ID	perfluoropolyether glycol content (mol %)	one-way scraping force (N)	the number of brine pinhole	the number of brine pinhole after winding
PU	0	3.52 ± 0.11	0	0
co-FPU20	20	3.25 ± 0.02	0	0
co-FPU50	50	3.56 ± 0.10	0	0
FPU100	100	3.21 ± 0.07	0	0

unilateral scraping force of the PU/co-FPU20/co-FPU50/FPU100-enameled wire coating is 3.52/3.25/3.56/3.21 N. According to GB/T 6109.23-2008,²⁵ the average scratch force of the enameled wire with a conductor diameter of 0.33 mm should be greater than 2.9 N, which can meet the requirements of Class 180 solderable polyurethane-enameled wire. The unilateral scraping forces of all enameled wires are not less than 3.21 N, which are higher than 2.9 N. This indicates that there is good adhesion between the coating and copper wire for all enameled wires. The introduction of fluorine does not affect the adhesion between the paint film and the copper wire. In the case of the brine pinhole test and the brine pinhole test after winding, the number of pinholes for all enameled wires is 0. This also indicates that the uniform paint film has good adhesion on the surface of copper wire. In addition, the flexibility of the paint film is also good, and the insulation coating layer does not crack even after the external force of winding.

As shown in Table 6, the breakdown voltages of the FPU-enameled wires are higher than that of PU. The introduction of

Table 6. Electrical Property of the FPU-Enameled Wires

sample ID	Perfluoropolyether glycol content (mol %)	breakdown voltage (V)
PU	0	2719.8 ± 57.0
co-FPU20	20	3368.0 ± 294.3
co-FPU50	50	3128.6 ± 18.8
FPU100	100	3169.6 ± 158.6

fluorine increases the breakdown voltage of enameled wires due to the stronger bond energy of the C–F bond than that of the C–C bond. In addition, the factors that affect the breakdown voltage also include the roundness of the paint film, the external impurities in the paint film, the degree of curing, and the thickness of the paint film.^{26,27} During the test, the eccentric state of the sample paint film, the technique, the strength, the speed of the kink, and the tightness of the two wires have effects on the breakdown voltage.^{28–30} Therefore, the breakdown voltages for the FPU-enameled wires have

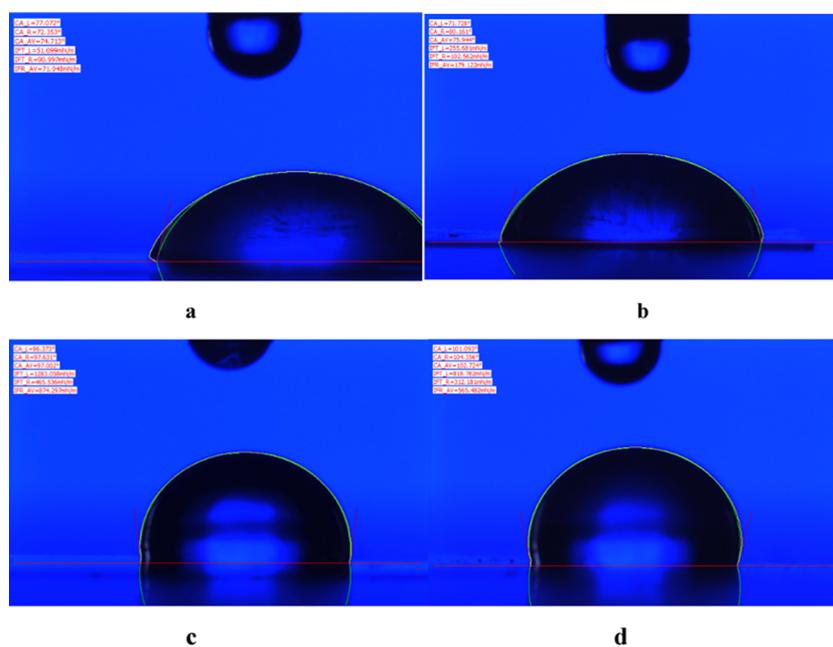


Figure 3. Static water contact angles: (a) PU, (b) co-FPU20, (c) co-FPU50, and (d) FPU100.

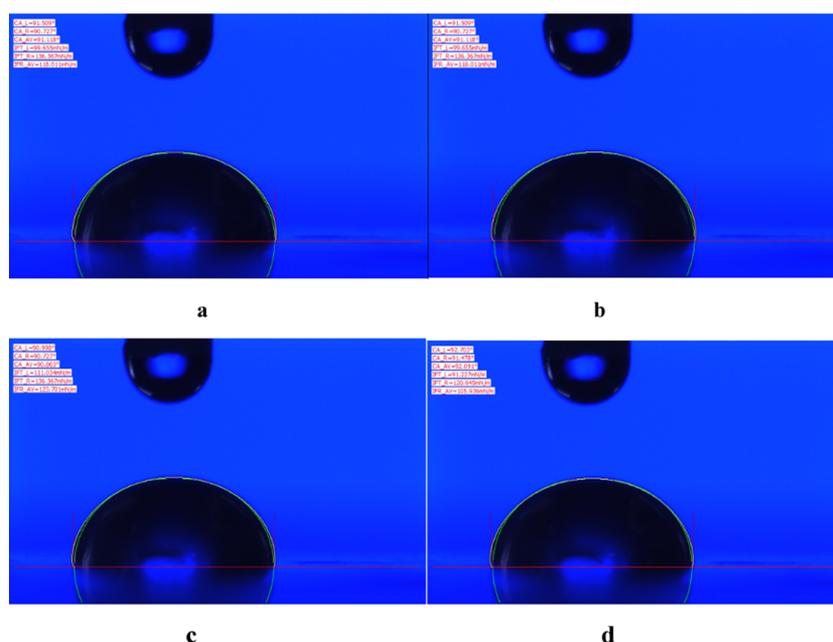


Figure 4. Static oil contact angles: (a) PU, (b) co-FPU20, (c) co-FPU50, and (d) FPU100.

some fluctuations. However, all FPU-enameled wires show greater breakdown voltages than the PU-enameled wires.

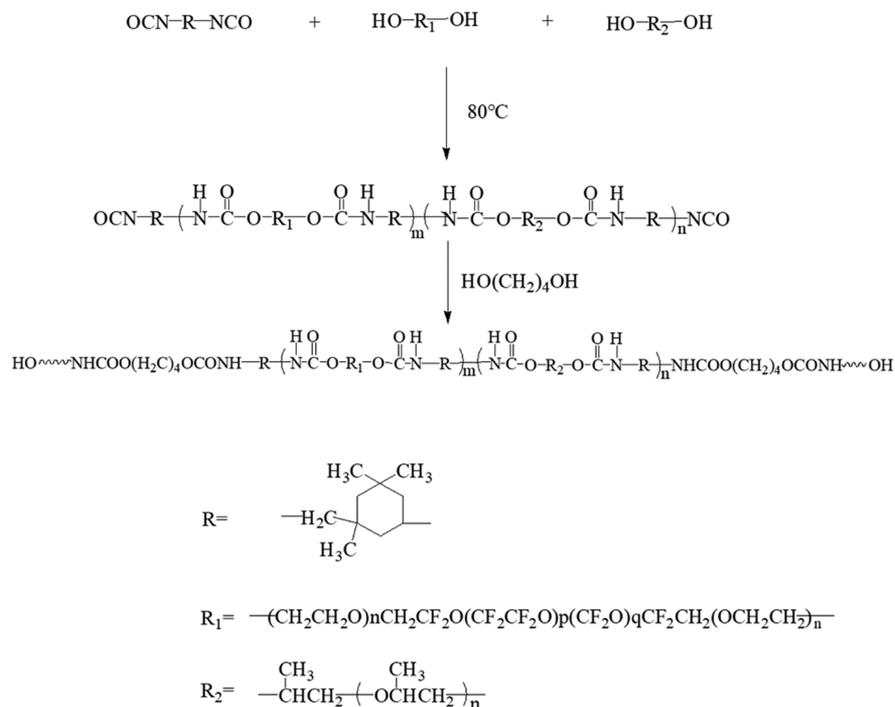
The contact angles between the samples and the deionized water are shown in Figure 3. The contact angles between the samples and ethylene glycol are shown in Figure 4. The contact angles of the samples are listed in Table 7. With the increase in the content of perfluoropolyether glycol in the polymer, the contact angle of water droplets increases from 74.7° for PU to 102.7° for FPU. The water contact angle of co-FPU20 is 75.9° due to the lower content of perfluoropolyether glycol. As the content of perfluoropolyether glycol increases to more than 50 mol %, the contact angles of water droplets are greater than 90° . With the gradual increase in the content of fluorine-containing segments, the enrichment of fluorine-containing

Table 7. Hydrophobic and Oil-Repellent Property of PU and FPU

	water contact angles (deg)	oil contact angles (deg)
PU	74.7	91.1
co-FPU20	75.9	91.1
co-FPU50	97.0	90.9
FPU100	102.7	92.1

segments on the surface is thermodynamically and kinetically favored by the low surface energy and high mobility of the fluorine-containing chain.³¹ Combined with the XPS results, more fluorine-containing groups are enriched on the surface

Scheme 1. Reaction Scheme to Synthesize FPU100, co-FPU50, co-FPU20, and PU



for FPU100. Therefore, FPU with more fluorine content exhibits stronger hydrophobicity.

The static oil contact angles of PU/co-FPU20/co-FPU50/FPU100 are 91.1/91.1/90.9/92.1°, respectively. PU itself has a certain degree of oil-repellent property. With the introduction of fluorine in the polymer, the contact angles are still greater than 90°, indicating a good oil-repellent property of the FPU samples. The polar carbamate groups in the PU molecular chain shows good oleophobicity to the oil phase of nonpolar or weakly polar solvents. PU is synthesized using PPG with a molecular weight of 400, the weight proportion of carbamate group content is relatively higher, therefore, the PU itself shows a certain degree of oleophobicity to the nonpolar oil phase. As the PPG content is decreased or the perfluoropolyether glycol content is increased for FPU, the weight proportion of the carbamate groups in FPU decreases due to the larger molecular weight of 900 for perfluoropolyether glycol. Thus, the increase in the fluorine content in combination with the decrease in the carbamate group content results in similar oil contact angles.

3. CONCLUSIONS

The fluorine element was introduced into polyurethane using perfluoropolyether glycol as starting materials, and fluorinated polyurethane with different fluorine content was successfully prepared by adjusting the ratio of perfluoropolyether glycol to PPG in the soft section. The contact angle test shows that FPU100 has hydrophobicity and oleophobicity. The enameled wires coated using the as-synthesized FPU100 show good adhesion between the uniform coating and copper wires. The number of pinholes in the brine is 0, the number of pinholes in the brine after winding is 0, and the one-way scratch resistance is 3.21 N. The breakdown voltage for FPU100-enameled wires is 3169.6 V. The friction coefficient of FPU100-enameled wires reaches 0.095, which is 36.2% lower than that of the fluorine-free enameled wires. The excellent properties of FPU100 cause

it to have broad application prospects in self-lubricating enameled wire coatings.

4. EXPERIMENTAL SECTION

4.1. Materials. Isophorone diisocyanate (IPDI), poly(propylene glycol) (PPG) ($M_n = 400$), dibutyltin dilaurate, dimethylbenzene, cresol, dibutylamine, and bromocresol green solution indicator were purchased from Macklin Co., Ltd. (Shanghai, China). Perfluoropolyether glycol (Fluorolink E10H, $M_n = 902$) was purchased from MoYan Chemical Co., Ltd. (Shanghai, China). 1,4-Butylene glycol (BDO) was purchased from Aladdin Co., Ltd. (Shanghai, China). Acetone was purchased from Tong Guang Co., Ltd. (Beijing, China).

4.2. Preparation of FPU. Perfluoropolyether glycol (18.04 g, 0.02 mol) and IPDI (14.0488 g, 0.0632 mol) and a certain amount of xylene were added in a four-necked flask with a mechanical stirrer, nitrogen inlet, and condenser. A 0.2 wt % dibutyltin dilaurate was used as a catalyst. The mixture was heated and kept at 80 °C with stirring for 3 h, and the FPU prepolymer was obtained. BDO (3.9759 g, 0.4412 mol) was then added into the flask to continue the reaction for 1.5 h. The as-obtained FPU was designated as FPU100.

As comparisons, polyurethane (PU) was synthesized using PPG and IPDI as starting materials. Fluorinated polyurethane copolymer with lower content of fluorine (co-FPU20) was synthesized by replacing 20 mol % of PPG with perfluoropolyether glycol. Fluorinated polyurethane copolymer with a higher content of fluorine (co-FPU50) was synthesized by replacing 50 mol % of PPG with perfluoropolyether glycol. The procedure of synthesizing FPU100, PU, co-FPU20, and co-FPU50 is illustrated in Scheme 1.

4.3. Preparation of Enameled Wires. The solution of PU, co-FPU20, co-FPU50, or FPU100 (15 wt %) in the mixture of phenol and xylene (volume ratio of 1:1) was used to coat copper wires with a diameter of 0.33 mm using a high-speed enameling machine, respectively.

4.4. Characterization. **4.4.1. Analysis of Fourier Transform Infrared Spectroscopy.** The Fourier transform infrared spectroscopy (FTIR) was recorded on a Thermo Scientific NICOLET IS10 with attenuated total reflection (ATR)-FTIR. The wavenumber range was 4000–500 cm^{-1} .

4.4.2. Molecular Weight of FPU. Gel permeation chromatograms (GPCs) were obtained on a Waters 410 instrument with tetrahydrofuran (THF) as an eluent at a flow rate of 1.00 mL/min.

4.4.3. Surface Composition of the FPU100 Film. The surface composition of the FPU100 film was examined by XPS instrument (Thermo Fisher, Thermo Scientific K-Alpha+) with monochromatic Al $K\alpha$ source (15 kV, KE = 1486.6 eV). The vacuum degree in the analysis chamber was 5×10^{-9} mbar. After the surface XPS was tested, argon ion etching was performed on it, and then XPS measurement was performed again.

4.4.4. Measurement of Contact Angles. The contact angles between the samples and deionized water were measured on a CAST3 type contact angle goniometer. The oil contact angle of fluorinated polyurethane with ethylene glycol as the oil phase was also tested.

4.4.5. Surface Properties of FPU-Enameled Wires. The friction coefficient of enameled wires was tested using the JMC-I type static friction tester according to GB/T 4074.3-2008.³² The static coefficient of friction (μ_s) was determined by measuring the inclining angle (α) of a plane at the moment when a block of 500 g began to slip on the track made from the wire specimen. One part of the wire specimen was straightened and then fixed on the inclining plane by means of the two posts and the two clamps constituting the sliding track. The other part of the wire specimen was mounted in a similar way on the sliding block. The sliding block with the wire specimen was then placed on the track of the plane to be inclined in such a way that the wire on the block and the wire on the plane were crossed at right angles at the point of contact.

The plane was then slowly inclined (approximately 1 degree per second) until the block started to slide down the track. At that moment, the angle of inclination (α) was read from the scale. The static coefficient of friction was calculated as follows

$$\mu_s = \tan \alpha$$

Five specimens were tested for every sample, and the average value was adopted.

4.4.6. Mechanical Properties of FPU-Enameled Wires. The brine pinhole was tested on a YZK-I type brine pinhole tester. The winding of enameled wires was carried out on a JR-100 type enameled wire winding tester. The adhesion between the paint film and the copper wire was tested using a WDG-400 unilateral scraper tester.

The brine pinhole was tested according to GB/T 4074.5-2008.³³ Take one sample and immerse it in 0.2% saline solution without undergoing any stretching or bending. The appropriate amount of 3% phenolphthalein alcohol solution indicator should be dropped into the saline solution first. During the test, the solution was connected to the positive electrode, the sample was connected to the negative electrode, a 12 V DC voltage was applied, and the number of pinholes generated on the sample within 1 min of the applied voltage was checked. A sample of about 6 m in length was taken, and the effective length of immersion in saline was 5 m. Five specimens were tested for every sample, and the average value was adopted.

The adhesion between the paint film and the copper wire was tested according to GB/T 4074.3-2008.³² The sample was wiped clean, put in the experimental equipment, and then fixed with a chuck and the support table was adjusted to contact the sample. The initial force applied to the scratching equipment should not be greater than 90% of the minimum scratching force specified by the relevant products; there should be a short circuit between the scraper needle and the conductor, and the short circuit point should be separated from the fixed fulcrum by 150–200 mm. The load-bearing scraping device slowly descended to the surface of the enameled wire and then began to scratch.

The scraping action would continue until the conductor was exposed and the machine was stopped. The test result was recorded. The experiment should be repeated twice in the same pattern, once from the original position of 120° and once from the original position of 240°. Test a sample and record three test values. The average value was taken as the average scratching force.

4.4.7. Electrical Properties of FPU-Enameled Wires. The breakdown voltage of enameled wires was tested using a QDS-15 kV voltage tester according to GB/T 4074.5-2008.³³ Twist was applied for a wire. A straight piece of wire, approximating 500 mm in length, with the insulation removed at both ends, was twisted back on itself for a distance of 125 mm with a load applied to the wire pair and with the number of twists as given. The loop at the end of the twisted section was cut; an AC voltage of 50–60 Hz was applied between the conductors of the wires. The test voltage was applied at zero and increased at 100 V/s, five specimens were tested, and breakage voltage of the wire was reported in the mean value of five tests.

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Notes

The authors declare no competing financial interest.

ACKNOWLEDGMENTS

This work was supported by the key projects of Industrial Core and Key Technologies in Zhuhai in 2018 (201821048001).

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