


Research on polyamide based self-lubricating composites: A review

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Abstract

Owing to its good tribological and mechanical properties, polyamide material was widely applied to prepare sliding mechanical parts. A lot of studies confirmed that adding different functional materials or surface modifications to polyamide could reduce the friction coefficient, improve wear resistance and reduce water absorption, so as to prolong the service life of polyamide parts. This paper summarized the common reinforcement methods of polyamide materials: blending, surface modification and filler. The reinforcing mechanism of different fillers and polymers, the effects of different methods on the tribological properties, mechanical properties and water absorption of polyamide composites, and the preparation methods of polyamide composites have been systematically discussed.

KEYWORDS

mechanical properties, polyamide composites, preparation methods, reinforcement methods, self-lubricating, tribological properties

1 | INTRODUCTION

Polyamide resin (PA, commonly known as nylon), was first developed as a product by W. H. Carothers in the 1930s. Due to the low density, high strength, wear and corrosion resistance, easy processing, and molding characteristics, polyamide has been widely used in many fields, such as electronics, machinery, chemicals, and aerospace equipment.^[1,2] However, the molecular structure of polyamide contains amide groups (—NHCO—), which are easily combined with water molecules to form hydrogen bonds, thus resulting in high water absorption, poor dimensional stability, and reduced performance of polyamide products.^[3] Although polyamide can replace

some metal and ceramic materials for the preparation of key components such as gears and bearings, most of these parts are used without lubrication, so polyamide self-lubricating materials must have good wear resistance and mechanical properties at the same time. In addition, polyamide is prone to failure for some mechanical parts due to its lower wear resistance under sliding conditions.^[4,5] Therefore, improving self-lubricating properties and mechanical strength and reducing water absorption are critical issues for polyamide materials' application in tribological fields.

Some polymers with good tribological properties and low water absorption (e.g., polyethylene, polyphenylene ether) are often blended with polyamide to perform

TABLE 1 Properties of composites after adding different polymers

Matrix	Reinforcement	Content	Coefficient of friction (COF)	Water absorption	Ref
PA46	HDPE	20 vol%	Decrease	-	[6]
PA6	UHMWPE	20 wt%	Decrease	Decrease	[7]
PA66	PPO	20 wt%	Decrease	-	[8]
PA6	PP	30 wt%	-	Decrease	[9]
PA6	PP/CF	30 wt%/5 wt%	-	Decrease	-
PA6	PBT	20 wt%	-	Decrease	[10]
PA66	PPS	30 vol%	Decrease	-	[11]
PA66	PPS/PTFE	30 vol%/30 vol%	Decrease	-	[12]

composites better. Modification by some physical or chemical methods can also give the polyamide surface new functions. With the development of nanotechnology, many low-dimensional materials have also been introduced into polyamide to enhance material performance. Nanoparticles, fibers, microcapsules and other lubricant fillers have significant effects on improving the mechanical properties of polyamide or reducing the friction coefficient. This paper summarizes the reinforcement methods and preparation processes of polyamide materials in recent years and explores the research status of polyamide based self-lubricating composite materials.

2 | PERFORMANCE ENHANCEMENT OF POLYAMIDE MATERIALS

2.1 | Co-blending enhancement

The selection of polymers with different properties according to different functional requirements and the flexible adjustment of the addition ratio of different polymers are the keys to preparing polyamide self-lubricating composites. As presented in Table 1, polymers with good hydrophobicity, high strength or low friction were blended with PA to enhance its properties.

Although polymers blended with polyamide can play a strengthening role, some polymers with high melt viscosity are difficult to combine with polyamide, and the blending materials are easy to peel off due to weak interfacial, which eventually leads to poor mechanical properties of the composites. Therefore, it is necessary to add an appropriate compatibilizer when blending the polymer with polyamide to promote bonding. Common polymers used for polyamide blending and corresponding compatibilizers were shown in Figure 1.

High-density polyethylene (HDPE) and UHMWPE have low production cost, good corrosion resistance and

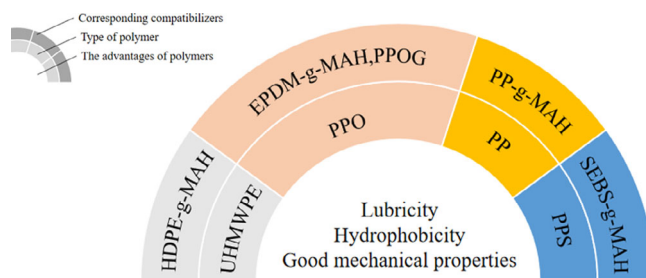


FIGURE 1 Commonly used polymers blended with polyamide and corresponding compatibilizers. EPDM-g-MAH, maleic anhydride graft ethylene propylene diene monomer rubber; HDPE-g-MAH, maleic anhydride grafted polyethylene; PP, polypropylene; PP-g-MAH, maleic anhydride grafted polypropylene; PPO, polyphenylene ether; PPOG, maleic anhydride grafted polyphenylene ether; PPS, polyphenylene sulfide; SEBS-g-MAH, styrene-butadiene-styrene grafted maleic anhydride; UHMWPE, ultra-high molecular weight polyethylene

low friction coefficient,^[13] and they are widely used in blending PA materials.^[14] Studies have shown that using HDPE-g-MAH as a compatibilizer could effectively solve the compatibility problem and improve the mechanical properties of the composite.^[15] When adding 20 vol% HDPE, the mechanical properties of the composites were relatively the best, and the wear rate could be kept low, which indicated that HDPE-g-MAH made the system more compatible and HDPE did not flake easily, which was conducive to the formation of a continuous and uniform transfer film.^[6,16,17] Adding HDPE to PA46 could improve the tribological properties of the composite. When the amount of HDPE added was 20 vol%, the friction coefficient of the composite was only 0.21, which was about 73% lower than that of pure PA46. However, as the HDPE content continued to increase, the friction coefficient would not continue to decrease but was slightly higher than that of pure HDPE, which was maintained at about 0.2, indicating that the effect of pure HDPE in reducing wear was limited. Liu et al.^[7] prepared

UHMWPE/PA6 (20 wt% UHMWPE) composites, whose water absorption was about 40% lower than that of pure PA6, and the tribological properties were significantly improved under the conditions of dry friction and oil in water lubrication; Although the two materials were well combined under the action of compatibilizer, the mechanical properties of the composites did not change significantly due to the softness of UHMWPE itself.

PPO is also one of the engineering plastics with good mechanical properties and dimensional stability. The addition of PPO to PA could reduce the water absorption and improve the tribological properties,^[18,19] which had essential applications in mechanical, chemical, and electronic fields.^[20,21] PPO is not easily compatible with PA and also requires styrene-maleic anhydride copolymer (SMA),^[22] maleic anhydride grafted polyethylene-octene copolymer (POE-g-MAH),^[23] polyphenylene ether grafted borate (B-g-PPO),^[24] and maleic anhydride grafted styrene-ethylene-butylene-styrene (SEBS-g-MAH)^[8] as compatibilizers to promote bonding.^[25,26] PPOG is the grafted product of PPO, which has similar physical properties to PPO. It can not only be used as a compatibilizer but also can be blended with PA as an excellent reinforcing material. The study found that compared with adding PPO, the mechanical properties of polyamide composites with PPOG were better, indicating that PPOG was more fully combined with the polyamide matrix. When short glass fiber (SGF) and PPO together reinforced PA6, the fibers not only fail to improve the mechanical properties of the composite, but their strength decrease compared to the raw material because of the poor bonding between the two and the polyamide matrix; this problem could be effectively solved by adding PPOG, and the tensile strength and bending strength of the composite were significantly improved when the additions of PPOG and SGF were 21 and 30 wt%, respectively.^[27]

PP is easy to process, has low density, and has certain hydrophobicity, adding PP to PA can also reduce the water absorption of composites. Aparna et al.^[9] used PP-g-MAH as a compatibilizer to explore the effect of different compatibilizer contents on composites' properties when the PP addition amount was 30 wt%. The study showed that the water absorption of the composites decreased by about 53.2% when no compatibilizer was added, and the water absorption of the composites decreased by about 60% when 4 wt% of PP-g-MAH was added. However, adding PP would reduce the mechanical properties of the composite material, and it was usually necessary to fill some other materials for reinforcement (such as fibers and carbon nanotubes [CNTs]).^[28,29] Polyethylene terephthalate (PBT) has a certain degree of hydrophobicity and maintains good mechanical

properties even after water absorption.^[30] Using epoxidized natural rubber as a compatibilizer could make PBT and PA6 bond better. When the PBT content was 20 wt%, the water absorption of the composite decreased by 31.2%, and the tensile strength and Young's modulus did not change significantly, but the hardness would decrease slightly.^[10] Such hydrophobic polymers are of great significance for developing polyamide based self-lubricating composites.

Polyphenylene sulfide (PPS) is a new type of special engineering plastic, with good mechanical properties, high-temperature resistance, corrosion resistance, not easy to absorb water, the application of excellent prospects, known as the sixth largest engineering plastic.^[31] SEBS-g-MAH as a compatibilizer could effectively solve the problem that PPS and PA were not easily mixed.^[32,33] Adding a specific content of PPS to PA66, the mechanical properties of the composite increased and then decreased with the increase of PPS content. When the PPS content was 30 vol%, the composite had the best mechanical properties; when the PPS content was higher than 40 vol%, the mechanical properties of the composite decreased, compared to pure PA66 due to the poor bonding of the two. The tribological performance of the composite was best at 20 vol% PPS, with a reduction in wear rate of ~25% compared to pure PA66, but there was no significant reduction in the coefficient of friction of the composite since PPS itself was not lubricious.^[11] The introduction of other polymers with better lubricity into the PA66/PPS system can further reduce the composite's friction coefficient and wear rate, but the mechanical properties of the composite are reduced with the addition of multiple polymers.^[12] As shown in Figure 2, the tribological properties of the composites were continuously improved with the addition of polytetrafluoroethylene (PTFE). When the content of PTFE was 30 vol%, the transfer film formed during friction was thinner and more uniform. At a load of 196 N and a sliding velocity of 0.42 m/s, the average coefficient of friction of the composites decreased by about 77%.

2.2 | Surface modification

Surface modification technology refers to improving specific properties of the base material or giving it new properties, and without affecting the properties of the base material itself. Commonly used modification methods include etching, deposition, laser, and surface grafting.^[34]

The principle of surface grafting is to change the properties of the substrate by connecting new structural units to the substrate molecules through chemical

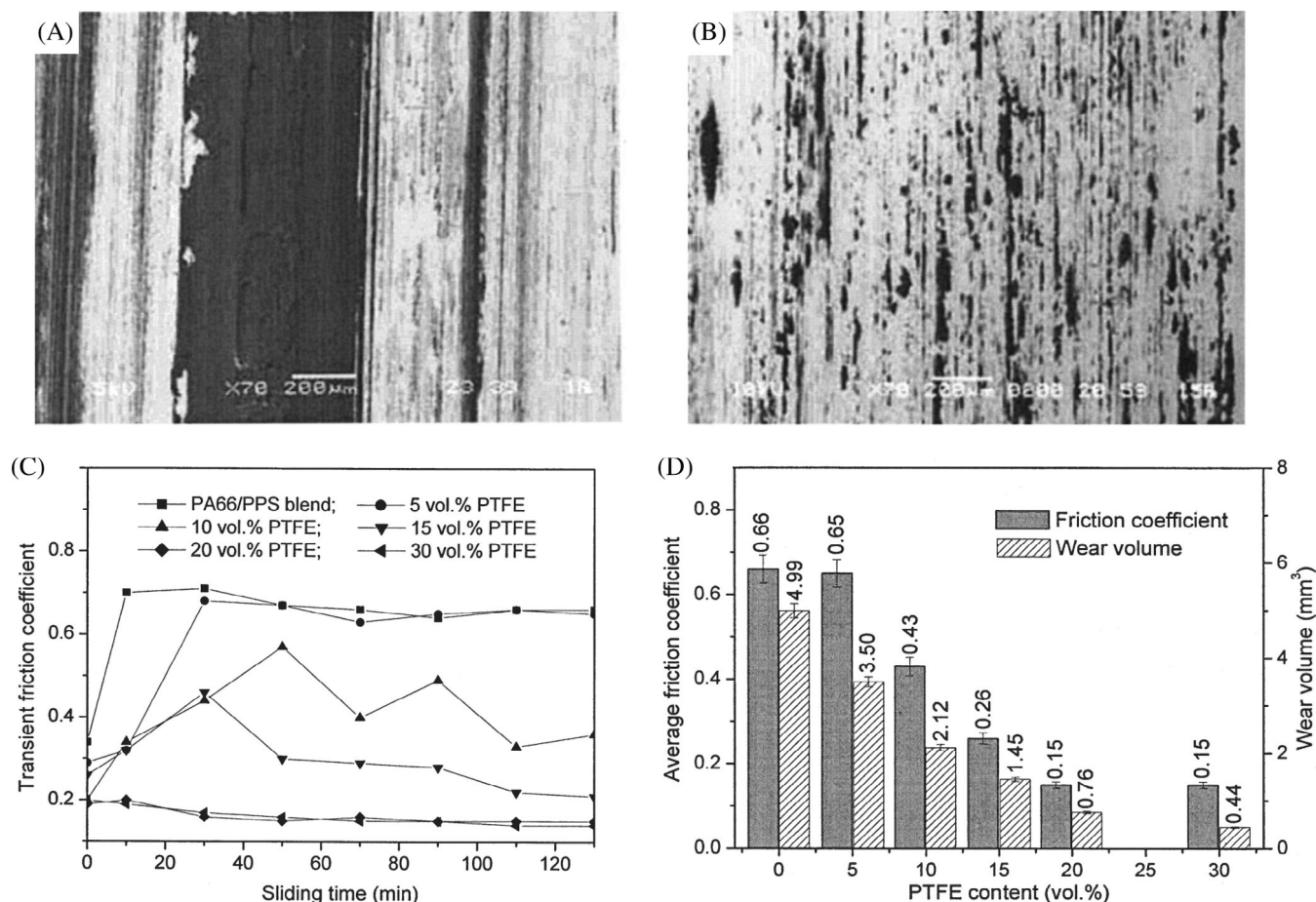


FIGURE 2 (A) Scanning electron microscope (SEM) micrograph of transfer film formed by PA66/PPS blend filled with 10 vol% PTFE. (B) SEM micrograph of transfer film formed by PA66/PPS blend filled with 30 vol% PTFE. (C) The real-time friction coefficient of PA66/PPS blends with different content of PTFE. (D) Friction coefficient and wear volume of PA66/PPS blends with different content of PTFE^[12]

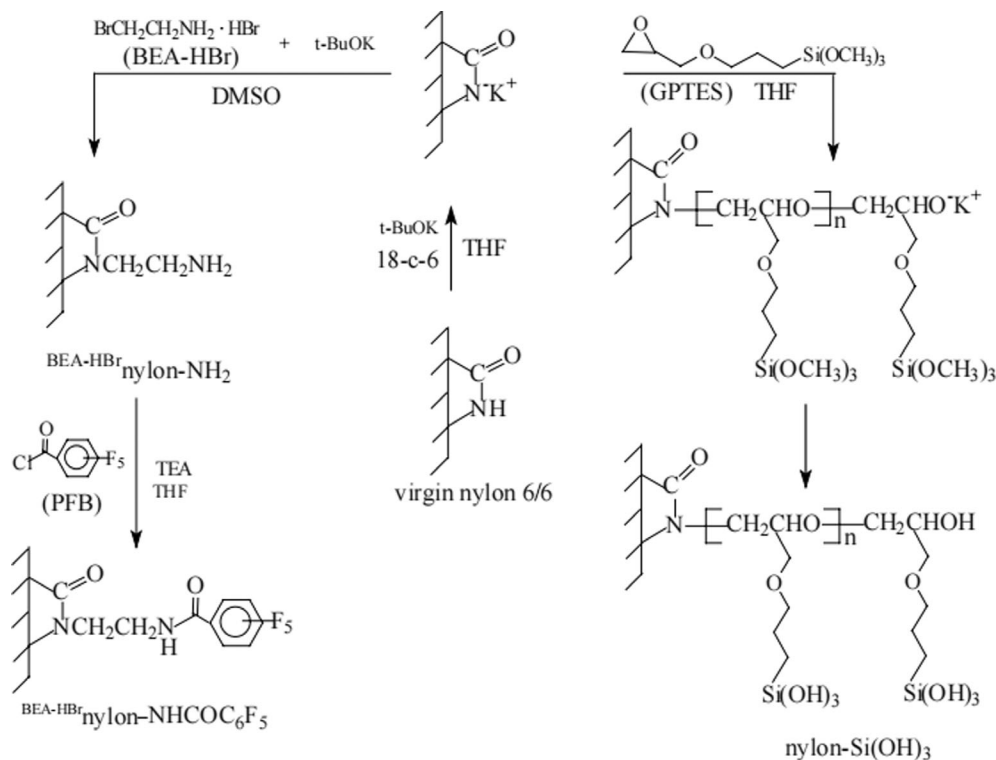
TABLE 2 Common grafting methods and characteristics

Method	Advantages	Disadvantages
Chemical grafting	Simple equipment and low cost	Long time and low grafting rate
UV-light grafting	Fast speed, low energy, no destruction of material	Uneven
Microwave grafting	Faster, more efficient, and more uniform	Only for polar monomers
Radiation grafting	Efficient and pollution-free	The small scope of application
Ozonation grafting	Simple equipment and low cost	

reactions, and this method also has a significant enhancement effect on PA. Standard grafting methods and characteristics were shown in Table 2. Generally, the factors that directly affect grafting were reaction temperature and modifier. The temperature and modifier content

could be controlled within a reasonable range according to the specific experimental needs to promote the grafting reaction.^[35] Glampedaki et al.^[36] successfully grafted chitosan-based hydrogels onto PA66 molecules and then tested the properties of PA66 fibers. It was found that the grafting of hydrogels did not affect the pore space and permeability of the fiber fabric but could effectively reduce the water absorption of the composites due to the hydrophobic nature of chitosan itself. Grafting poly-N-isopropyl acrylamide (NIPAM) onto PA66 followed by passivation could also improve the polyamide properties. The water contact angle of the material increased significantly when the temperature reached 35°C, and the material became hydrophobic from the original hydrophilic.^[37] Most grafting modifications to polyamide had been done to make it hydrophobic, flame retardant,^[38] and antibacterial,^[39] and there were few studies on grafting modification of polyamide for tribological properties. Since the essence of graft modification is to replace the groups on the polyamide molecular chain with new structures, in most cases, PA6 and PA66 with more

FIGURE 3 Principles of different modification methods^[41]



amine end groups are modified to reduce the water absorption of composite materials. Block materials cannot be grafted with high efficiency, so grafting modification of fibers or films is more effective, but this method is less applicable.

In addition to surface grafting, it is also a method for polyamide modification by coating the polyamide surface or constructing functional surfaces. Coating a layer of nano-SiO₂ and nano-ZnO composite film on the polyamide surface could improve the hydrophobicity of the material. In order to make the film's hydrophobic effect better, the composite film could be modified with sodium stearate (SS). When the concentration of SS was 50 mmol/L, the polyamide composites could obtain super hydrophobicity capacity.^[40] Polyamide could also be modified by chemical reactions, and amine groups could be formed by reacting with polyamide using 2-bromoethylamine hydrobromide (BEA-HBr), thus allowing polyamide to gain hydrophobic capacity; It was also possible to use (3-glycidoxypropyl) triethoxysilane (GPTES) to graft first, and then prepared a hydrophobic surface, and the specific process of different chemical reactions was shown in Figure 3.^[41] The study found that the modification effect of BEA-HBr was better than that of GPSES, and the modified polyamide surface water contact angle was larger.

Plasma treatment was also an effective means of surface modification, commonly using argon or nitrogen ion immersion. The treatment caused cross-linking of the polymer molecular chains and enabled the formation of new microstructures on the surface, which could play a

significant role in improving the properties of the polymer. This method requires some experimental equipment and was more expensive, but it ensures clean and intact materials and could be used for most materials.^[42–45]

Kalácska et al.^[46] used nitrogen ions with a flux of $3 \times 10^{17}/\text{cm}^2$ to modify PA6, and after the treatment, the amide matrix in PA6 decomposed, the N element content of the material increased, and the C and O element content decreased. This also reduced the contact angle of PA6 due to the increase in the nitrogen-containing functional groups and affected the tribological properties of PA6. They compared the tribological properties of modified PA6 under both dry friction and water-lubricated friction. As shown in Figure 4, under dry friction conditions, the friction coefficient of modified PA6 increased first and then decreased with the increase of load, and finally remained at about 0.5. The specific wear rate of the sample was slightly reduced relative to the raw material at low loads; Under water lubrication conditions, the friction coefficient fluctuated. As the load increased, the friction coefficient increased, and the specific wear rate of modified PA6 decreased significantly. As shown in Figure 4E, the transfer film of nitrogen ion-treated PA6 was prone to adhesion during friction, so the coefficient of friction was not significantly reduced.

2.3 | Reinforced with fillers

Filling materials with high strength, hardness, or self-lubricating properties into the polyamide matrix is also a

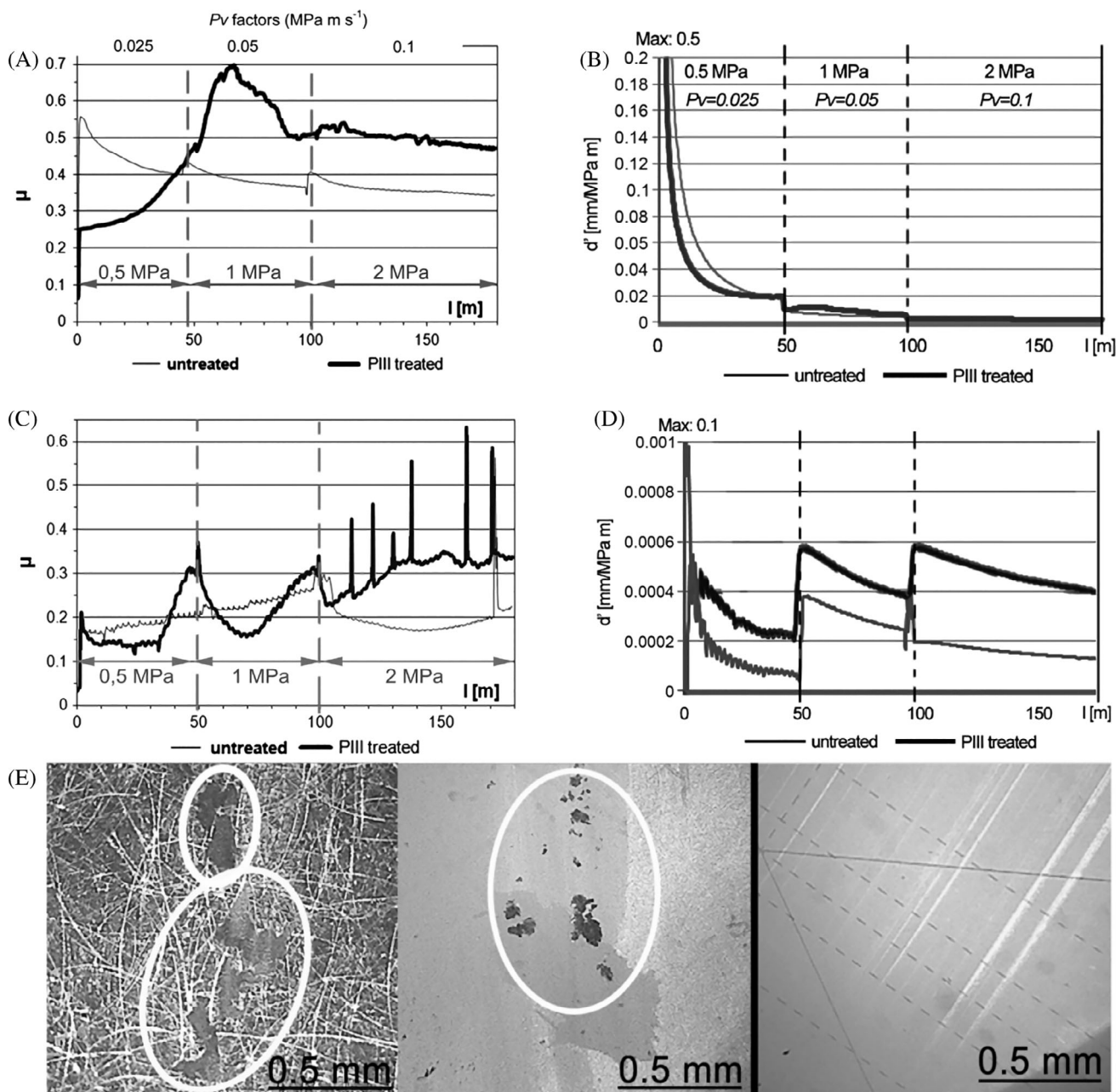


FIGURE 4 (A) Pin-on-disc test under dry conditions ($v = 0.05$ m/s); (B) specific wear rate under dry conditions; (C) pin-on-disc test under water lubricated conditions ($v = 0.05$ m/s); (D) specific wear rate under water lubricated conditions; (E) optical micrographs of the contact surfaces after dry sliding test. (Left: the surface of steel disc, middle: the surface of treated PA6, right: the surface of untreated PA6)^[46]

common means of reinforcement and these materials range from the micron to the nanoscale with a wealth of selectivity. Typical polyamide reinforced fillers were shown in Figure 5 and related research was summarized in Table 3.

2.3.1 | Lubricating oil/grease

Lubricating oil/grease could be used as reinforcing materials to fill polyamide to enhance its lubricating

properties. Adding hydraulic lubricating oil to monomer casting (MC) polyamide could reduce the coefficient of friction of composite materials: the friction coefficient of the composite material with 8 wt% lubricating oil was only 0.16 at the load of 200 N and the rotation speed of 200 r/min; however, compared with pure MC polyamide, the tensile strength, flexural strength and compressive strength of the composites decreased by 41%, 37%, and 70% respectively, owing to the oil droplets dispersing in the matrix to form a porous structure (as shown in

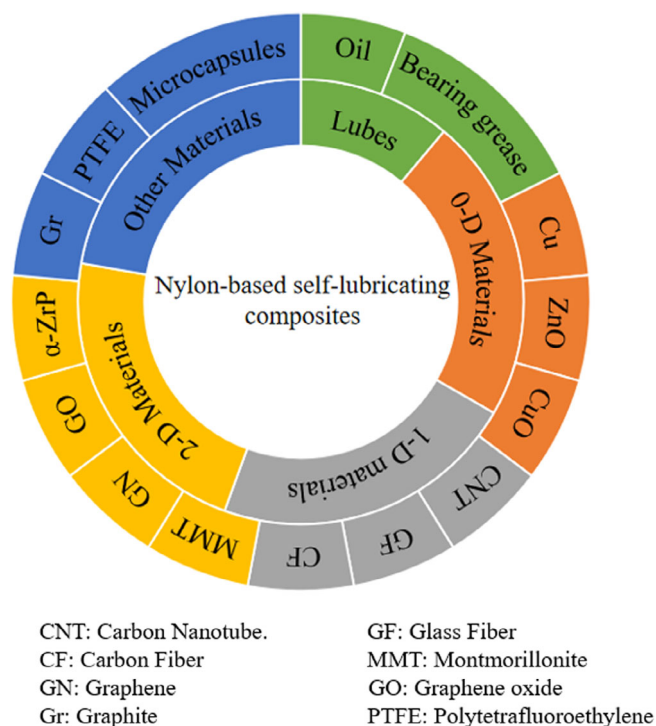


FIGURE 5 Commonly used fillers for reinforced polyamide.

Figure 6), which affected the loading capacity of the material.^[47] Kang et al.^[67] compared the mechanical and tribological properties of lubricating oil-filled and wax-filled PA6 composites. It was found that there was no significant difference in the mechanical properties between the two composites and the mechanical properties of the composites became worse with the increase of lubricant content; when the lubricating oil content was 6 wt%, the friction coefficient of the composite was the lowest. A large number of studies have shown that although polyamide filled with lubricating oil/grease has a good effect in reducing the friction coefficient, the mechanical properties and wear rate of the composites will have obvious defects. So far, it is still a challenge to prepare self-lubricating composites with better comprehensive properties by filling polyamide with lubricating oil.

2.3.2 | Zero-dimensional materials

Due to its small size, large specific surface area, and unique volume effect, zero-dimensional materials show great advantages in improving resin properties.^[68] Some soft metal particles (such as gold and silver) are easy to shear and have good thermal conductivity. Under some conditions, they can be used as good lubricants to reduce friction.^[69–71] However, there are relatively few kinds of metals that can be used as lubricants and the cost is

relatively high. Therefore, nano metal compounds such as metal oxides, sulfides, and nitrides are increasingly added to polymers to improve material properties.

Metal oxides exhibit lower shear strength and surface adhesion due to their higher ionic potential energy,^[72,73] making them widely used as filler materials. Metal oxides commonly added to polyamide include titanium dioxide nanoparticles (TiO_2),^[74] zinc oxide nanoparticles (ZnO),^[75] and aluminum oxide nanoparticles (Al_2O_3).^[76] It was found that adding nano- Al_2O_3 to the PA66 matrix could reduce the friction coefficient and wear rate of the composites. Two weight percent Al_2O_3 had the best effect on enhancing the tribological properties of PA66: the friction coefficient could be reduced to about 0.2, and the wear rate was also reduced at the load of 80 N and the sliding speed of 6.5 m/s.^[48] Although metal oxides have a good reinforcing effect, they are inorganic nanoparticles, which have weak bonding with polyamide matrix and are easy to peel and fail in the process of friction. In order to make the interfacial bonding between metal oxides and polyamide matrix better, Ambrosio et al.^[49] modified the surface of TiO_2 with organic reagents (APTMS) and then explored the mechanical and tribological properties of $\text{TiO}_2/\text{PA11}$ composites. It was found that the modified TiO_2 had a better enhancement effect on the mechanical properties and wear resistance of PA11, and the wear amount decreased from 144 to 43 mg.

2.3.3 | One-dimensional materials

Adding CNTs with high strength, high hardness, good conductivity, and thermal conductivity can improve the mechanical properties and wear resistance of polyamide.^[77] Meng et al.^[50] incorporated CNT into a PA6 matrix and compared the performance of the composites under dry friction and water lubrication; with the addition of CNT, the mechanical properties of PA6 could be effectively improved, and the water absorption of the composite reduced. Due to the good thermal conductivity of CNT, the temperature rise in the friction region could be decreased, thus reducing the adhesion of the material, which could result in a lower coefficient of friction of the composite. The reinforcing effect of CNT with different lengths on PA66 was also quite different.^[78] When the temperature of the friction pair was lower than 110°C , the short CNT had a better effect on reducing the wear rate of the composite material. When the temperature was higher than 110°C , the enhancement of the material's surface by the long CNT was more pronounced, which could effectively reduce the wear rate. At the low temperature, the softening of the composite material was not obvious, the temperature had a low effect on the

TABLE 3 Various polyamides, fillers, and fillers' effects

Matrix	Filler	Content	Effect on COF	Effect on wear rate	Effect on mechanical properties	Ref
MC PA	Hydraulic oil	8 wt%	Decrease	-	Increased tensile strength, bending strength, and compression strength	[47]
PA66	Nano-Al ₂ O ₃	2 wt%	Decrease	Decrease	Increased tensile strength, flexural strength, and compressive strength	[48]
PA11	TiO ₂ (APTMS modified)	4 wt%	-	Decrease	Increased elastic modulus and hardness, decreased yield strain	[49]
PA6	CNT	1 wt%	Decrease	Decrease	Increased tensile strength and Young' s modulus, decreased elongation at break	[50]
PA12	GF	30 wt%	Decrease	Decrease	Increased tensile strength and Young' s modulus, decreased elongation at break	[51]
PA6	SBF	5 wt%	Decrease	Decrease	Increased tensile strength and tensile modulus	[52]
PA1010	CF (Nitric acid oxidized)	20 wt%	Decrease	Decrease	Increased tensile strength and hardness	[53]
PA6	CF (Ozone modified)	20 vol%	Decrease	Decrease	Increased interfacial shear strength	[54]
MC PA	GN (PEG disperse)	0.5 wt%	Decrease	Decrease	Increased tensile strength and tensile stress	[55]
MC PA	GO (Organic amine modified)	0.05 wt%	Decrease	Decrease	Increased tensile strength and shore hardness	[56]
PA66	MMT (Oganic modified)	5 wt%	-	-	Increased tensile and flexural strengths, decreased impact strength	[57]
PA66	α -ZrP (Obtained at 3 M H ₃ PO ₄)	1 wt%	Decrease	Decrease	Increased tensile modulus, decreased tensile strength	[58]
PA6	LDH	3 wt%	-	-	Increased elasticity modulus, decreased tensile stress and elongation at break	[59]
PA6	Gr	20 wt%	Decrease	Decrease	Increased tensile strength, decreased elongation at break	[60]
PA6	Wax	4 wt%	-	Decrease	Increased tensile strength, decreased tensile modulus	[61]
PA66	PTFE	20 wt%	Decrease	Decrease	-	[62]
MC PA	Microcapsule (lube base oil)	20 wt%	Decrease	Decrease	Decrease in hardness	[63]
PA11	Microcapsule (PAO)	50 wt%	Decrease	Decrease	Decrease in hardness	[64]
PA6	CF/PTFE	15 wt %/20 wt%	Decrease	Decrease	-	[65]
PA1010	CF/MoS ₂	10 wt %/10 wt%	Decrease	Decrease	-	[66]

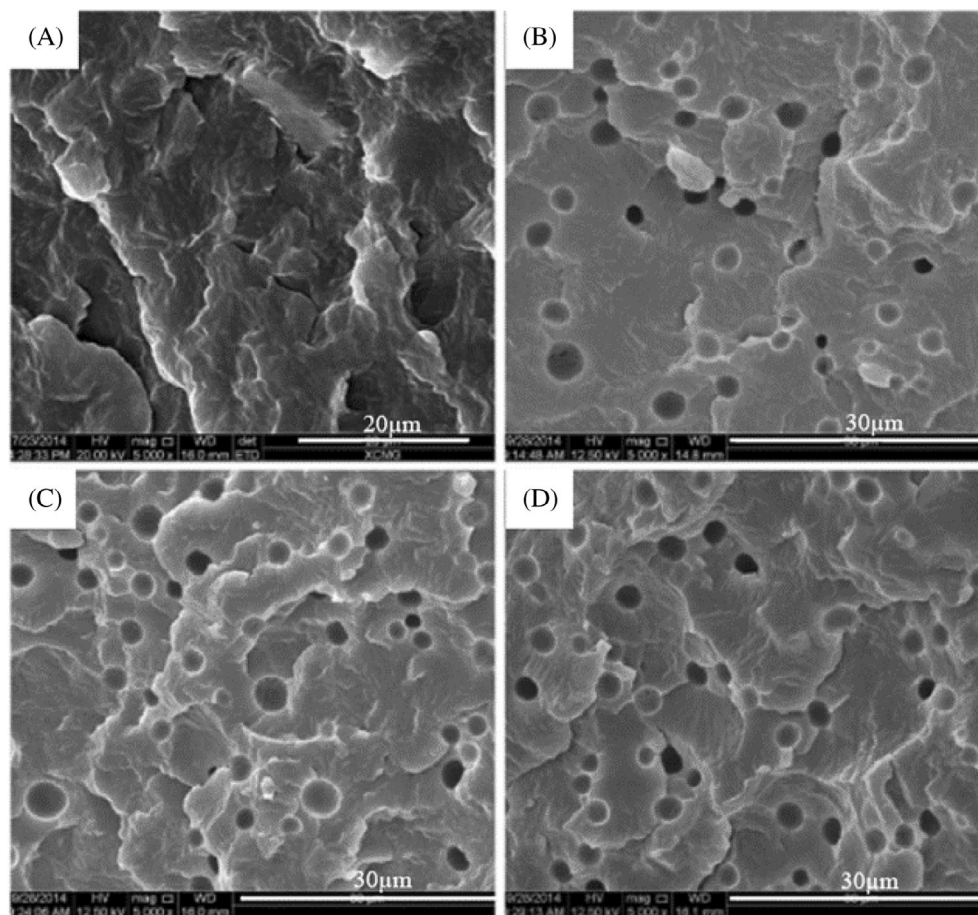
tribological properties, and the short CNT could better disperse the load and improve the wear resistance. At the high temperature, long CNT could maximize the friction performance and had better heat dissipation performance.

Calcium silicate whiskers had excellent mechanical properties and could be dispersed uniformly in the polyamide matrix without any modification or modifications. The addition of calcium silicate whiskers to PA1012 could reduce the water absorption of the composite. With the increase of whisker content, the tensile and flexural strength of the composites increased, but the impact

strength decreased; The best performance of the composite was achieved when the calcium silicate whisker content was 20 wt%, the water absorption rate of 1 h could be reduced to about 0.4%. Tensile strength and flexural strength increased by about 10% and 50% respectively compared to pure PA1012.^[79]

Polyamide could be reinforced by adding some fiber materials, such as carbon fiber (CF), glass fiber (GF), and basalt fiber (BF). Due to their low water absorption, good wear resistance and mechanical properties, these fiber materials could transfer and disperse load in the friction process, so as to weaken the cutting effect and

FIGURE 6 Scanning electron microscope (SEM) morphology of monomer casting (MC) polyamide composites filled with different contents of hydraulic oil (A) 0, (B) 2 wt%, (C) 4 wt%, (D) 8 wt%^[47]



significantly improve the tribological properties of polyamide. It was found that adding 25%–35% GF to PA12 matrix could effectively improve its tribological properties.^[51] Raajeshkrishna et al.^[52] added short basalt (SBF) to PA6 resin, and the friction coefficient and specific wear rate of polyamide composites decreased significantly with the increase of SBF content; When the load was less than 20 N and the SBF content was 5 wt%, the friction coefficient of the composites decreased with the increase of the load. The tensile strength, flexural strength and impact strength of SBF/PA6 composites containing 5% SBF were increased by about 60%, 57%, and 85% respectively.

Although the different fibers provided some reinforcement and wear reduction, the filled fibers were exposed, broken, and peeled off with friction due to the poor bonding of the fibers to the polyamide resin, which could affect the formation of the transfer film and reduce the tribological properties of the polyamide composite. Therefore, improving the interfacial bonding properties of fibers is essential to enhancing the self-lubricating properties of composites. Commonly used methods include coupling agent treatment, plasma treatment, acid, and alkali etching, and fiber surface coating.^[28,80–82]

Related studies compared the bonding ability of CF with PA6 after plasma treatment, low-temperature treatment, and nitric acid oxidation, respectively, and found that the surface of CF after using nitric acid oxidation was rougher and had the best bonding with the substrate, as shown in Figure 7.^[83] By adding the nitric acid oxidation-treated CF to PA1010, the wear resistance of the composite was significantly improved in both the dry and wet states of the composite, and the addition of CF also resulted in better dimensional stability of the material.^[53] Nie et al.^[54] compared the surface modification effects of different oxidation methods on carbon fibers, and the study showed that the properties of carbon fibers oxidized by ozone were better than those of ordinary high-temperature oxidized carbon fibers. The friction coefficient and specific wear rate of polyamide composites reinforced with modified carbon fibers were significantly reduced.

In addition to CNTs and various fibers, erosive nanotubes and titanate nanotubes could be used as modified materials to strengthen polyamide materials.^[84] These nanomaterials could reduce the water absorption of polyamide and improve the mechanical properties, but the enhancement effect on tribological properties was limited.^[85]

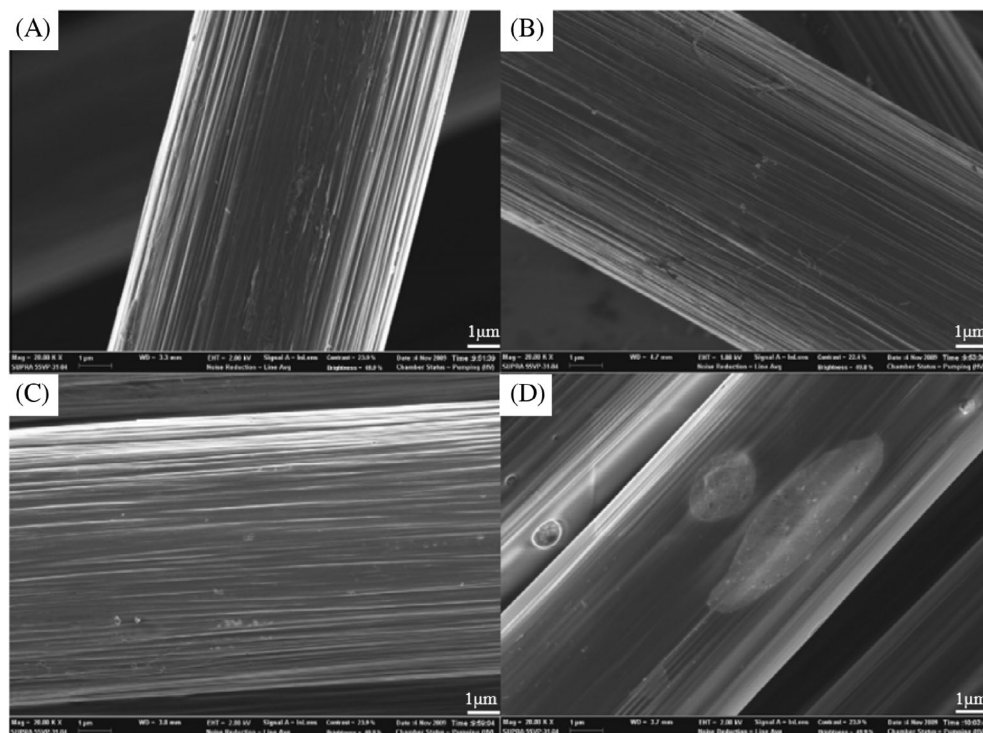


FIGURE 7 Scanning electron microscope (SEM) picture of CF after different methods of processing (A) untreated, (B) plasma treated, (C) cryogenically treated, (D) nitric acid treated^[83]

2.3.4 | Two-dimensional materials

Two-dimensional materials such as graphene (GN), graphene oxide (GO), montmorillonite (MMT), and mica have layered structure, which is easily sheared and exhibit good lubricating properties, are commonly used as filling materials for polyamide.^[86]

Due to the small size and energy instability of two-dimensional materials, they are prone to agglomeration under van der Waals or electrostatic forces, and to prevent this phenomenon, some chemical reactions or mechanical actions are generally required to improve dispersion.^[87] Li et al.^[55] used polyethylene glycol (PEG) as a dispersant to add GN to PA66. The study showed that 0.5 wt% GN could improve the mechanical and tribological properties of the composite; The content of GN could further reduce the friction coefficient and wear amount of the composite, but its mechanical properties would also decrease slightly. The composites prepared with dispersants had better properties, which showed that GN was uniformly dispersed in the matrix and could play a positive role in reducing wear. Oxidation of GN to form GO was also an effective method to prevent agglomeration, and studies have shown that GO had better adhesion to the matrix due to its higher specific surface area and the ability to react with polymer molecules chemically.^[88] Pan et al.^[56] added organic amine-modified GO to MC polyamide and found that a small amount of modified GO had a significant enhancement effect. When the modified GO content was 0.05 wt%, the composites' friction coefficient and specific wear rate

were reduced by 92% and 14% compared to pure MC polyamide.

MMT and mica are inorganic two-dimensional nanomaterials, which are essentially layered silicate. Since clay materials are poorly compatible with polymers, they need to be modified to enhance their bonding with the matrix.^[89] Since the lamellar structure of MMT could prevent the entry of water molecules when MMT was added to PA, the water absorption of the composite decreased with the increase of MMT content.^[90] Mohanty et al.^[57] treated sodium-based montmorillonite (Na-MMT) with different modifiers to compare the enhancement effect of different modifiers on PA6. The study found that the modification effect of methyl tallow bis-2-hydroxyethyl quaternary ammonium salt was better than that of octadecylamine and hexadecyl trimethyl ammonium bromide, and adding 5 wt% modified MMT could make the composites obtain the best mechanical properties. Adding nano clay material and polypropylene into PA6 could significantly improve the tribological properties of the composites, and the friction coefficient decreased from 0.5 to 0.25 at the load of 50 N and the sliding speed of 5 m/s, while the tensile strength and tensile modulus of the composites also increased.^[91]

Alpha-layered zirconium phosphate (α -ZrP) is a new two-dimensional material with easy preparation, good thermal stability, certain lubricity, and wide application. α -ZrP has smaller layer spacing than MMT, only 0.76 nm.^[92,93] Sun et al.^[58] incorporated α -ZrP nanosheets of different scales into PA66 and micron-level

α -ZrP reinforcement was more effective, with the friction coefficient and wear rate of the composite decreasing by 43% and 60% compared to pure PA66 at an addition of 1 wt%. There is relatively little research on ZrP/PA composite self-lubricating materials, and more related studies are expected to be carried out in the future. Bimetallic hydroxides (LDH) is a layered compound composed of metal hydroxide. As an additive, LDH could improve the mechanical properties and heat resistance of materials.^[94–96] The research has shown that the addition of 3 wt% LDH could reduce the water absorption of PA6 material and improve its mechanical properties because the addition of LDH would reduce the molecular gap of PA and make it more difficult for acrylamide to combine with water molecules.^[59]

2.3.5 | Other fillers

In addition to filling with lubricants and some low-dimensional materials, some fillers with excellent lubricating properties or mechanical properties can also play a role in lubrication and wear reduction. Graphite is a kind of solid lubricant with anisotropic properties, and the lamellar structure is more easily sheared to form a transfer film during friction.^[96,97] Graphite did not weaken the mechanical properties as significantly as wax.^[61] The addition of graphite to PA6 could improve the composites' wear resistance and mechanical properties, with the best tribological and mechanical properties when the graphite content was 20 wt%^[60] and the best wear resistance when the graphite filling was 25 wt%.^[98] Polytetrafluoroethylene (PTFE) can play a role in wear reduction because its molecular chain is smooth and easy to form a transfer film, different from the laminate material.^[99,100] The addition of PTFE particles to PA6 could reduce the coefficient of friction of PA6 and improve wear resistance.^[62,101] In contrast to filled lubricants, the use of PTFE-filled polymers resulted in the better formation of continuous transfer films near the friction substrate and more stable tribological properties.^[102]

The unique “core-shell” structure of microcapsules allows the lubricant to be better dispersed in the matrix and the lubricant to escape from the wall under the action of shear during friction, thus improving the tribological properties of the composite.^[103–106] Gao et al.^[63] added lubricant-containing microcapsules to MC PA6 to make a composite coating, and the coefficient of friction and mechanical properties of the material decreased as the microcapsule content continued to increase. When the microcapsule content was too high, the composite did not bind tightly enough, and the specific wear rate increased instead; a microcapsule content of 20 wt% was most

reasonable, and the friction coefficient and wear rate decreased by \sim 57% and 92%. Poly alpha-olefin synthetic oil (PAO) is a synthetic lubricant with excellent lubricating properties. Filling PAO-containing microcapsules into PA11 could improve its tribological properties, and the friction coefficient of PA11 could be reduced to as low as 0.1 after adding 50 wt% oil-containing microcapsules.^[64] Although microcapsules could effectively solve the problem of the dispersion of lubricating oil in the resin matrix, the addition of microcapsules would reduce the mechanical properties of the composite material, and if the microcapsules amount was too large, it would cause more significant wear.

2.3.6 | Multi-fillers

Although using a single reinforcing material can reduce the coefficient of friction or water absorption of polyamide, it may encounter problems such as reduced mechanical properties and weakened wear resistance. Modifying polyamide with different types of reinforcing materials can realize complementary properties, so that polyamide can show better comprehensive properties. Due to its high strength, high hardness, and high wear resistance, the fiber is very suitable for reinforcing polyamide materials together with other fillers.

By adding fibers and lubricating materials together to polyamide, the comprehensive performance of the composite material can be improved, the mechanical properties of the composite material can be improved, such as improving the mechanical properties of the composite, reducing the wear rate, and ensuring its low friction coefficient.^[65] Wang et al.^[66] studied the effects of MOS_2 and CF on the properties of PA1010 materials, and found that although the simple addition of MOS_2 could reduce the friction coefficient of the composite from 0.63 to 0.4, the wear rate was about 20% higher than that of the raw material. After adding 10 wt% CF and 10 wt% MOS_2 together to PA1010, under the conditions of 100 N load and 0.42 m/s sliding speed, the friction coefficient of the composite material was 0.25, the wear rate was reduced by about 95%, and the formed transfer film was more continuous and uniform.

Fibers can also be used with polymers, microcapsules, and so forth, to reinforce polyamide. Ran et al.^[107] added carbon fibers to PA46 using polyurethane cladding treatment (BCF) and PPO, and with PA46 wt%: PPO wt% = 9:1, the wear volume of the composite decreased continuously with increasing BCF; when the BCF content was 40 wt%, the friction coefficient of the composite decreased by about 40%, while the tensile strength and flexural strength also increased to 3.5 times and 2.7 times of pure PA46. Suresha et al.^[108] compared the mechanical and tribological properties of PP/PA66, Gr/PP/PA66,

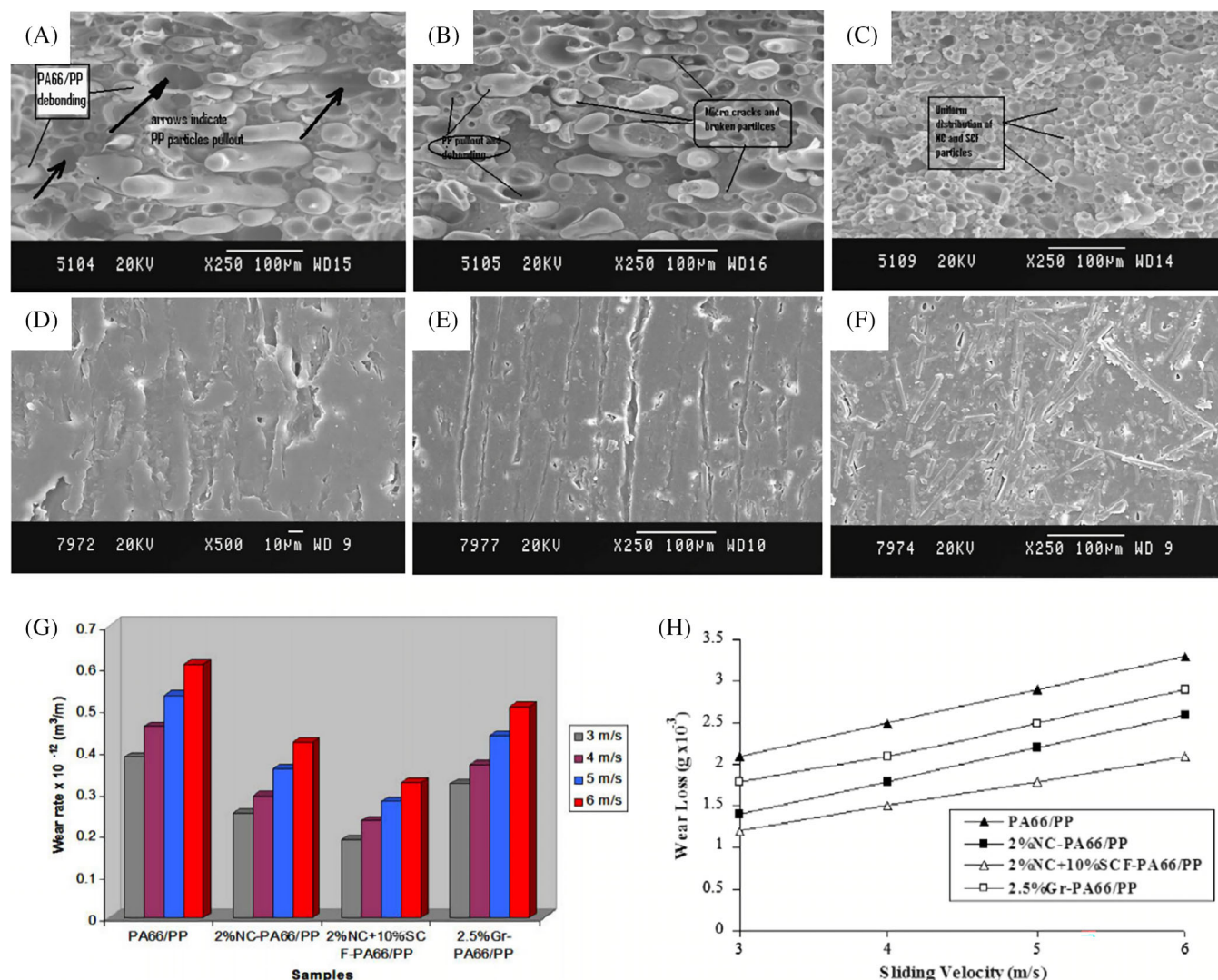


FIGURE 8 Top: Scanning electron micrograph of tensile fracture, (A) PA66/PP composite, (B) 2 wt% NC filled PA66/PP nanocomposite, (C) 2 wt% NC + 10 wt% SCF filled PA66/PP nanocomposite; middle: scanning electron micrograph of different composites at 80 N, 6000 m, and 6 m/s, (D) PA66/PP composite, (E) 2 wt% wt NC filled PA66/PP nanocomposite, (F) 2 wt% NC + 10 wt% SCF filled PA66/PP nanocomposite; bottom: wear rate and wear loss of particulate filled PA66/PP nanocomposites at a load of 80 N and a sliding distance of 6000 m, (G) wear rate, (H) wear loss^[108]

nano-clay/PP/PA66, and nano-clay/PP/SCF/PA66 composites. The study found that when polyamide was reinforced with one or two materials, the mechanical properties of the composites decreased to varying degrees. As shown in Figure 8, when polyamide was reinforced by the three together, there would be neither obvious fiber stripping marks nor pores on the fracture surface, which indicated that the filler was well combined with the matrix, and the mechanical properties of the composites were very close to those of pure PA66 (a-c); The multi-component reinforcement reduced the thermal deformation during friction, and the SCF could reduce the wear scar produced by the composite material during friction (d-f), so the composite material showed excellent

tribological properties. At a load of 80 N and a sliding velocity of 6 m/s, the friction coefficient was only 0.2.

3 | PREPARATION OF POLYAMIDE BASED SELF-LUBRICATING COMPOSITES

As mentioned earlier, different polymers and fillers can be used to strengthen polyamide. How to mix and combine these materials with polyamide matrix is a key problem. Melt blending and in situ polymerization are common preparation methods of polyamide matrix composites.

3.1 | Melt blending

The melt blending method is not only suitable for filling some polymers in polyamide, but also for most fillers to modify polyamide. It has the advantages of simple operation, low cost, and mass production. Solid raw materials are transported by screw shear force after melting at a high temperature of 260–300°C.

When fibers or particles were filled with polyamide, if the stress inside the agglomerates was sheared, the agglomerates would be broken and uniformly dispersed in the polyamide matrix. However, when the filler particles or fibers were too small, the shear force had a limited dispersing effect on the agglomerates, and the filler could not be uniformly dispersed. Therefore, it is necessary to increase the shear force or change the shear method to prevent agglomeration.^[109] Compared to single-screw extruders, twin-screw extruders are more homogeneous in mixing and are more widely used in blending filler-reinforced polyamide and polymers. Shon et al.^[110] found that, when using fibers to fill polymers, the anisotropic twin-screw extruder allowed for a more homogeneous mix but tends to disrupt the shape of the fibers and caused them to break. They also compared the effects of different extrusion equipment on the PP/PA6 blending system and found that the use of twin-screw extruders gave better dispersion than standard mixers or kneaders. Anisotropic twin-screw extruders better prevented

agglomeration and more uniform polymer dispersion than co-rotating twin-screw extruders.^[111]

3.2 | In situ polymerization

In situ polymerization has been widely used in polyamide based nanocomposites. First, the monomer of polyamide and nanomaterials were evenly mixed by ultrasonic dispersion or mechanical stirring, and then the appropriate initiator was selected for polymerization at the appropriate temperature and pressure. During the in situ polymerization process, the nanomaterials were involved in the overall reaction and could be fully integrated with the polyamide molecules to enhance their interfacial bonding while avoiding the agglomeration of the nanomaterials. Although agglomeration can be effectively avoided, the polymerized product generally needs to be processed by a die or an extruder to obtain an ideal sample. The application of in situ polymerization is diverse, whether it is organic nanomaterials or inorganic nanomaterials filled polyamide, this method can be used for synthesis; At the same time, the in situ polymerization method is not limited by the material dimension and is suitable for the preparation of most polyamide based nanocomposites. SiO₂,^[112] single-walled carbon nanotubes (SWNT),^[113] multi-walled carbon nanotubes (MWNT),^[114] and Gr^[115] could be effectively combined

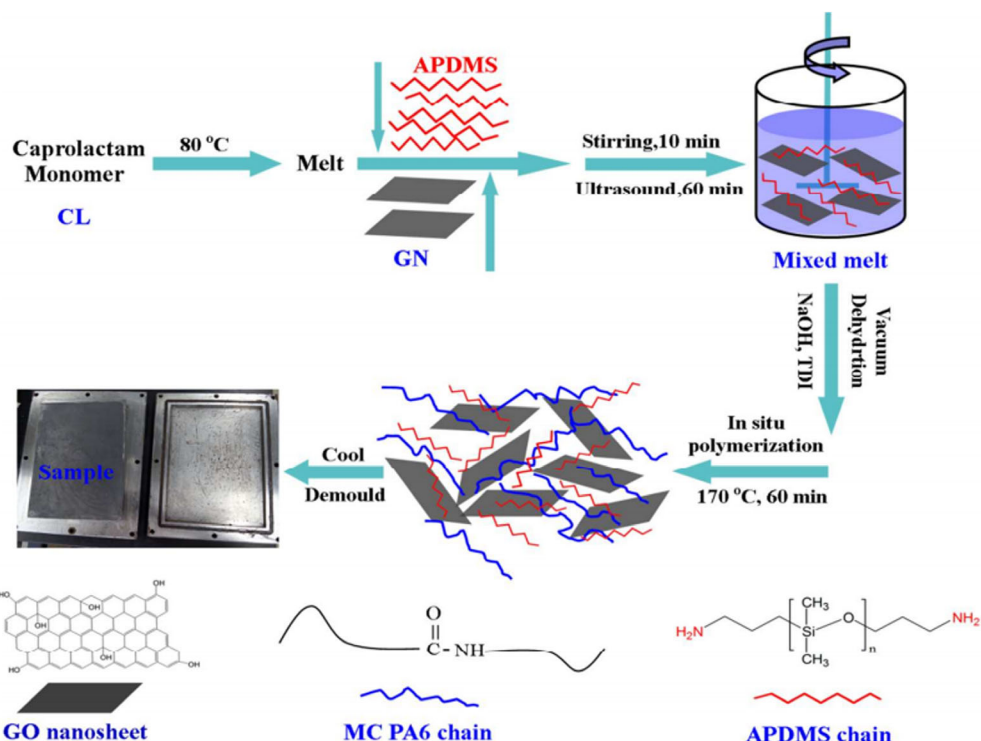


FIGURE 9 Process for the preparation of MC PA6/GN-APDMS nanocomposites^[119]

with the polyamide matrix by in situ polymerization, and the mechanical properties of the composites were significantly improved.

3.3 | Interpolation composite method

The PA6/nano-clay composites were first prepared by the intercalation method by Okada et al.^[116] Unlike in situ polymerization, the intercalation method is only suitable for the preparation of layered material-reinforced polyamide composites. First, the lamellar structure of the layered material was disrupted and dispersed into nanoscale units, which were then uniformly dispersed in the polymer matrix to achieve a good combination of polymer and layered nanomaterial. With material science and technology development, some scholars have proposed the in situ intercalation polymerization method. The polymer monomers were polymerized after the layered nanomaterials were uniformly dispersed. This method had broader applicability and better dispersion of fillers.^[117,118] As shown in Figure 9, it was the preparation process of MC PA6/GN-3-aminopropyl-terminated polydimethylsiloxane (APDMS) nanocomposite.^[119] Compared with ordinary melt blending and in situ polymerization, GN participated in the polymerization after exfoliation and dispersion, which made the dispersion uniform and the reaction sufficient. The composite material not only improved the mechanical properties but also reduced the friction coefficient and specific wear rate by 81% and 48%, respectively, compared with pure MC PA6.

4 | SUMMARY AND OUTLOOK

This paper reviewed the research progress of polyamide self-lubricating composites in recent years, summarized the different strengthening methods, mechanisms, and different preparation methods. So far, the development of polyamide based self-lubricating composites with good comprehensive properties for practical engineering is still facing great challenges. Combined with the current research status of polyamide composites, the existing problems and development directions are put forward as follows:

The addition of appropriate polymers and fillers could improve the self-lubricating ability of polyamide. However, some materials could affect the mechanical properties and wear resistance of composite materials while reducing the coefficient of friction of polyamide. When adding polymers, compatibility with the polyamide matrix should also be considered, and a suitable compatibilizer should be used to promote the bonding.

There were many surface modification methods of polyamide, but there were few studies on tribological properties, and surface grafting and lamination were limited by the chemical composition and morphology of the matrix. Plasma treatment had a limited effect on improving the tribological properties of polyamide. Exploring more effective methods to improve the self-lubricating ability of polyamide materials remains a significant challenge.

Various new nanomaterials with excellent performance can be introduced into polyamide composites for modification, but filling nanomaterials should also prevent agglomeration. Polyamide based nanocomposites have great prospects, and a variety of functional materials jointly enhance polyamide is also a current research hotspot.

In preparing polyamide based self-lubricating composites, different processes should be selected according to different fillers and reinforcement mechanisms. At the same time, polyamide should be prevented from absorbing water and oxidation when it was treated.

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DATA AVAILABILITY STATEMENT

Since this is a review, data sharing is not applicable to this article as no new data were created or analyzed in this study. Cited data are available from the original authors.

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