Progress in ionizing radiation resistance modification of polymer materials

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Abstract: With the wide application of polymer materials, much attention has been paid to the modification methods of polymer materials with high-energy radiation resistance to satisfy special environment, such as nuclear industry, space technology, medical equipment. In this review, progress in ionizing radiation resistance modification of polymer materials is introduced in depth and different modification methods are compared. Finally, future perspectives of this field are discussed.

1. Introduction

Polymers are widely used in medical, aerospace, nuclear power plants and nuclear weapons because of the advantage of low density, specific strength, good electrical insulation and corrosion resistance, and easy processing. However, compared with metals and inorganic materials, polymers are more easily damaged by ion radiation [1-2]. After the primary reaction of high energy radiation, such as the excitation and ionization of outer electrons and formation of free radicals, cross-linking and scission of polymer molecular chains further take place. Cross-linking reaction and scission reaction occur simultaneously in most polymers after irradiation, but generally one reaction is dominant and the other reaction can be ignored [3-5]. After radiation, the mechanical properties, thermal stability and chemical stability of most polymers become worse, coloring, and electrical conductivity increases, resulting in functional failure of materials [6-7]. Therefore, improving the service life and reliability of polymers under ionizing radiation environment has attracted people's attention. At present, aromatic compounds, antioxidant and nano materials are introduced in polymer matrix materials to improve the radiation resistance.

2. Introduction of aromatic compound

Because the stable benzene conjugated structure can well disperse radiation energy, the efficiency of collision-induced attenuation process is improved and the molecular chain fracture of local C-C bond caused by excitation is reduced. Thus, aromatic groups show high radiation stability. It is generally believed that the more aromatic groups in the molecular chain, the better its radiation resistance.
2.1. Physical modification

There are two main methods to modify polymer materials by introducing aromatic compounds: physical modification and chemical modification. Physical modification mainly involves blending small aromatic compounds with polymer matrix materials. The physical modification method is simple in process, but has compatibility problems. With the increase of additive amount, the mechanical properties of materials often decline. Moreover, the radiation energy transfer is an intermolecular behavior, and the protection efficiency is relatively low, so physical modification is characterized as external protection[11-12].

By addition of nine kinds of aromatic compounds such as benzene, naphthalene and biphenyl to \( n \)-dodecane, Tabuse et al.[13] found that the yield of scission product decreases continuously with the increase of the aromaticity and concentration of the additive after analyzing the final product of \( \gamma \) radiation. The reactivity of additives and transient intermediates was analyzed by pulse radiolysis. It was found that the reactivity becomes lively with higher degree of aromaticity of additives. The authors think that the reaction of additives and intermediate species is an important factor to inhibit radiation effect.

Chen et al.[14] blended styrene-ethylene-butylene-styrene block copolymer (SEBS) with polystyrene (PS) to improve the radiation resistance of SEBS elastomer. On this basis, diphenylacetylene, biphenyl, anthracene and pyrene were added into the SEBS/PS blend system, and it was found that aromatic compounds improve the mechanical, thermal and dielectric properties of the blends after \( \gamma \) radiation. Among them, pyrene with the highest aromatic properties shows the best protective effect. The authors suggest that energy transfer may be more important than the reaction of additives and free radicals in radiation protection.

Using ethylene–propylene–diene terpolymer (EPDM) as matrix, phenyl vinyl methyl rubber as anti-radiation additive and magnesium hydroxide as flame retardant, the irradiation resistant, halogen-free, low smoke generating and flame retardant EPDM insulating material for cable was prepared by Wang et al.[15]. The best formula was obtained by orthogonal test, and the flame retardant properties, mechanical properties and radiation resistance of the obtained composite materials are good.

2.2. Chemical modification

The blending of aromatic compounds into polymer matrix usually has poor compatibility and dispersion, and these compounds are often highly carcinogenic substances, harmful to human body. In order to improve the compatibility at molecular level, aromatic functional groups are introduced into the molecular chain by grafting or copolymerization through structural design, which can play a better radiation resistance effect[16-17].

Alexander et al.[18] prepared isobutene and styrene copolymers with styrene content of 20%, 50% and 80%, and the study showed that the styrene unit under \( \gamma \) radiation undergoes cross-linking reaction, which could provide radiation protection for adjacent isobutene units and prevent the scission of the chain. The authors believe that styrene units are randomly distributed, so even when the content is increased to 80%, there are still some isobutene units that are not effectively protected.

Delides et al.[19] studied the \( \gamma \) radiation effects of polydimethylidyphenylsioxane (PDMDPS) copolymers and polymethylsiloxane (PDMS) and PDMDPS mixtures, and found that the radiation-resistant protective coefficient increased exponentially with the increase of phenyl content. At a content of less than 10%, the protection range of phenyl is 6 monomer units of dimethylsiloxane (DMS), while when the content exceeds 10%, the protection range of phenyl is reduced to two or three monomer units of DMS.

Feng et al.[20-21] synthesized polyvinylsilicone oil with condensed aromatics (called C1 gum) by Diels-Alder reaction of polyvinylsilicone oil andacenaphthenecyclone, and then added it to silicone rubber. In the study, the radiation rate was 150 kGy/min and the radiation dose was 300, 500 and 1000
kGy, respectively. It was found that C1 gum was successfully incorporated to the cross-linking network of silicone rubber. Therefore, compared with the physical modification of 9,10-diphenylanthracene, the condensed aromatics rings on C1 gum could better transfer the radiation energy and play an anti-radiation protection effect of internal protection. On this basis, the team used tetraphenylphenyltrioethoxysilane (TPHPHS) and TPHPHS and hexamethyldisilazane (MMN) to simultaneously modify the surface of silica nanoreinforcers, and then studied the radiation protection effect of modified silica nanoreinforcers on silicone rubber. The results show that TPHPHS and MMN modified silica nanoreinforcers (TPHMTS) can not only improve the radiation resistance of silicone rubber, but also improve mechanical properties. As internal protection, TPHMTS has better radiation protection effect than C1 gum because its large conjugated groups are widely dispersed.

3. Introduction of antioxidants
Antioxidants can not only prevent the aging of polymer materials, but also improve the radiation resistance of materials through hydrogen supply, decomposition of hydrogen peroxide, scavenging free radicals and passivation of metal. According to the molecular structure, antioxidants can be divided into amine antioxidants, phenolic antioxidants, phosphate ester antioxidants, thio-ester antioxidants and compound antioxidants[22-24].

Aquino et al.[25-26] mixed 0.3% wt of hindered amine antioxidant Tinuvin 622 in poly(methyl methacrylate) (PMMA) and analyzed performance under γ radiation at room temperature in air. When the radiation dose is higher than 10 kGy, the yellowing index of PMMA and PMMA-622 increases with the dose, but the increase of PMMA-622 is much smaller, and the reduction of adhesive molecular weight of PMMA -622 is also smaller, which is conducive to maintaining the material's mechanics. With the addition of Tinuvin 622, the thermal decomposition reaction activation of the material increases by 60 kJ/mol and the number of free radicals is reduced.

Cai et al.[27] added four kinds of antioxidant Irganox 1010, Irganox 1076, Tinuvin 770 and Chimassorb 944 in polypropylene (PP) respectively and compared their modification effects under electron beam radiation. Compared with pure PP, the blending system with antioxidants has higher crystallinity and impact strength, but lower thermal stability, tensile strength and elongation at break. Except Irganox 1010, the other three antioxidants improve yellowing resistance of PP after radiation, and yellowing index of Tinuvin 770 is the lowest.

Wang et al.[28-29] studied the combination effect and mechanism of hindered amine antioxidant PDS (copolymer of 4-(methacrylic acid)-2,2,6,6-tetramethylpiperidol ester and styrene) with other types of antioxidants in PP under γ radiation. The retention of elongation at break was used as the index to evaluate the effect of radiation resistance modification. It was found that the synergistic effect is obvious when PDS is added together with hindered phenol antioxidants Irganox 1010 and Irganox 1076, but not with Irganox 1098. PDS and phosphorus-containing antioxidant Irgafos 168, sulfur-containing antioxidant Irganox PS 802 or aromatic amine-based antioxidant 4010 all produced certain synergies, of which 4010 is best. The co-modification effect of PDS and compound antioxidant is not as good as that of single antioxidant, and sometimes there may be counter-synergistic effect. The author believes that the synergistic effect of PDS and most antioxidants is attributed to the effectively removes of hydroperoxides and peroxyradicals generated in PP after radiation.

Many natural compounds, such as micro algae, vitamin E, rosemary, β-carotene, have the effect of scavenging and inhibiting free radicals and are known as biological antioxidants. In recent years, with the increasing attention paid to environmental protection and health, the potential of biological antioxidants for ionizing radiation modification in polymers has received more and more attention[30-32].

Zaharescu et al.[33-35] added Chlorella vulgaris and Spirulina platensis powders to the EPDM substrate to prepare eco-friendly materials, and found that thermal and irradiation stability of the composite materials are improved, of which Spirulina platensis is better. Under γ radiation, the number of carbonyl radicals and hydroxyl radicals in the mixing system decreased significantly because the polyphenols in Chlorella vulgaris and Spirulina platensis can effectively absorb radiation
energy and improve the activation energy of oxidation reaction. At the same time, the team also used gallnut acid and rosemary to prepare an environmentally friendly EPDM that can withstand high energy radiation. In addition, the authors added rosemary to improve the ionizing radiation resistance of low density polyethylene (LDPE) and ultra-high molecular weight polyethylene (UHMWPE), and the results showed that rosemary can block the reaction of free radicals and oxygen, and improve oxidation activation.

4. Introduction of nanomaterials
As a typical mesoscopic system, nanomaterials have surface effect, small size effect, macroscopic quantum tunneling effect and excellent optical, thermal, electrical, magnetic, mechanical and chemical properties. Nanomaterials can not only absorb a large amount of energy, but also be a highly efficient radical scavenger[36-37]. At present, carbon-based nanomaterials, layered silicate and polydopamine are mainly used to improve the radiation resistance of polymers.

4.1. Carbon-based nanomaterials
The radiation protection effect of carbon-based nanomaterials is mainly realized through the following free radical scavenging mechanism: radical adduct formation at sp2 carbon sites; electron transfer; hydrogen donation from functional groups; chelation of transition metal ions and inhibition of Fenton-based radical generation[38-41].

4.1.1. Graphene
Graphene is a new material with sp2 hybridized carbon atoms tightly stacked into a single layer of two-dimensional honeycomb lattice structure, with excellent optical, electrical and mechanic properties. Graphene has excellent free radical scavenging efficiency due to large surface area and conjugate structure, and has broad application prospects in radiation-resistant materials. Studies have shown that few-layer graphene (FLG) has the strongest ability to scavenge free radicals, followed by reduced graphene oxide (RGO), and graphene oxide (GO) is inferior[42].

Xia et al.[43] prepared GO modified epoxy resin radiation resistant coating. When the amount of addition is 0.25 wt%, GO can effectively reduce the content of free radicals in the coating. However, GO is unevenly dispersed and agglomeration phenomenon and hole-shaped defects appear as the content continues to increase, which increase the diffusion efficiency of oxygen and the concentration of free radicals. Therefore, the degree of dispersion of graphene in the polymer material matrix plays a key role in improving the radiation resistance, and the small amount of addition also ensures the economy of the final product.

Figure 1. Schematic of the mechanism for the improvement of the radiation resistance of PMMA/RGO nanocomposites

In order to improve the dispersibility of graphene, Wu et al.[44] prepared RGO modified PMMA composites by a modified latex blending approach. The free radical scavenging ability of RGO and the physical insulation ability of the flake structure to oxygen diffusion have a synergistic effect, which greatly reduces the molecular chain cross-linking and scission behaviors of RGO/PMMA
nanocomposites under high and low doses of γ radiation, so the mechanical properties and thermal stability of composite materials are improved (Figure 1).

Wu’s research team also added RGO and the antioxidant (AO1010) to PP resin, and found that RGO has a good protective effect in low-dose radiation and AO1010 is more suitable for high-dose radiation environments. When 0.5 wt% AO1010 and 1.0 wt% RGO are added at the same time, the mechanical properties and thermal stability of the nanocomposite improve even more. The authors believe that AO1010 and RGO play an additive role[45].

4.1.2. Carbon nanotube
Carbon nanotubes can be seen as curled graphene sheets, with large surface area, large aspect ratio, excellent mechanical properties, electrical conductivity, heat transfer performance and chemical stability, and are widely used in the preparation of polymer nanocomposites with excellent properties[46-47]. Multi-walled carbon nanotubes are relatively cheap, so they are often used to improve the radiation resistance of polymers.

Martinez-Morlanes et al.[48-49] modified UHMWPE with multi-walled carbon nanotubes, and found that the overall performance of the nanocomposites is improved. Electron spin resonance detection showed that the number of free radicals produced by γ radiation decreases with increase of carbon nanotubes, and the reduction of propylene radicals is the largest. The mechanical performance test showed that the Young's modulus of the nanocomposite increases by up to 73% after radiation, and the toughness basically unchanged. Thermogravimetric analysis showed that the thermal decomposition temperature of the coating increases.

In order to further improve the compatibility of multi-walled carbon nanotubes with the polymer matrix, Chang et al.[50] prepared multi-walled carbon nanotubes with carboxyl functional groups on the surface (c-MWCNTs), and then used to improve the radiation resistance of epoxy resin. It was found that the carboxyl functional group reacted with the amino and hydroxyl groups of the epoxy resin to make c-MWCNTs participate in the formation of the epoxy resin cross-linked grid, thereby significantly improving the radiation resistance and comprehensive performance of the composite coating. And the optimal addition amount of c-MWCNTs is 0.75%.

4.2. Layered silicate
Zhang et al.[51] added organophilic montmorillonite (OMMT) to the styrene-butadiene-styrene block copolymer (SBS) matrix to prepare SBS/OMMT nanocomposites. When subjected to γ radiation under oxygen atmosphere, OMMT can effectively prevent the decrease of the storage modulus of SBS/OMMT nanocomposites. The author believes that this is because OMMT can not only absorb a large amount of radiation energy, but also generate a large number of free radicals to react with the free radicals generated by the SBS matrix after being radiated, preventing further decomposition of the molecular chain, thereby improving the radiation resistance.

The γ radiation effects of EPDM, EPDM/clay nanocomposites and its conventional composites with pristine clay were studied by Ahmadi et al[52]. The tensile strength of EPDM increases under low-dose radiation, but decreases after the dose continue to increase. XRD and TEM results show that the organic sodium montmorillonite is uniformly dispersed in the EPDM matrix, forming an interpenetrating network structure. Therefore, after radiation of 500 kGy, the tensile strength of nanocomposites is 51% and 78% higher than that of conventional composites and unfilled EPDM, respectively.

Tiwari et al.[53] used a melt extrusion method to mix organically modified clay with poly(vinylidene fluoride) (PVDF) to prepare radiation-resistant nanocomposites (NCs), and studied the radiation resistance performance under different fluxes of the bombardment of high-energy swift heavy ions (SHI). Under higher bombardment, the crystallinity and melting temperature of pure PVDF decreases significantly, while the modified NCs remain unchanged. Atomic force microscope observation of the surface morphology shows that the pitting size and degradation of pure PVDF greatly increases after the flux exceeded 1011 ions/cm², while NCs containing layered organically
modified clay have almost no defects and degradation, indicating that the organically modified clay can effectively improve the radiation resistance.

4.3. Polydopamine
Although graphene, carbon nanotubes and nanoclays can effectively improve the radiation resistance of polymers, as inorganic materials, they have poor compatibility with polymer matrix materials and often require organic modification. Moreover, it is difficult for graphene and carbon nanotubes to be uniformly dispersed in the matrix material, and strong acid or alkali solutions are often used in the preparation process, resulting in serious pollution and high cost[54]. The preparation process of polydopamine (PDA) is simple and environmentally friendly. It has good compatibility with polymers due to a large number of functional groups on the surface and almost the same structure as natural melanin gives PDA excellent free radical scavenging ability[55-57].

Lv et al.[58] prepared polydopamine and polyurethane (PU) blended nanocomposites by melt casting. Under 200 kGy radiation, the cross section of PU shows obvious cracks, but no cracks in the nanocomposites with only 1% of polydopamine nanoparticles (PDAPs). And the maximum decomposition rate temperature increases greatly by 50°C. The tensile strength also increases from 1.48 MPa (pure PU) to 5.48 MPa (5% PDAPs), and the elongation at break of pure PU decreases by 33.9% while increases by 19.3% with addition of 5% PDAPs. Figure 2 demonstrates the mechanism of enhanced radiation resistance of PU after incorporation with PDAPs.

Chen et al.[59-60] blended polydopamine with PU, and studied the effect of PDA on the microphase structure and properties of PU under γ radiation. The results show that PDA can protect the soft and hard segments of PU and increase the storage modulus and glass transition temperature of PU elastomers. The tensile strengths of PU and PDA/PU both decrease with increase of radiation dose, but PDA nanospheres can effectively alleviate the downward trend, especially when the radiation dose is less than 100 kGy. The authors also blended polydopamine-modified graphene (PDA-GNP) with PU to improve radiation resistance, and found that PDA-GNP is connected to the PU backbone through a large number of covalent bonds. As the content of PDA-GNP increases, the number of the hard segment microphases of PU increases and size decreases. By adding only 0.5% of the content, the tensile strength, elongation at break and toughness of the composite increase by 313%, 16%, and 279%, respectively.

5. Conclusion
The ionizing radiation-resistant modified polymers can reliably serve in high-energy radiation environments and have good application prospects in many fields such as medical treatment, space technology, and nuclear industry. In the future, the development of antioxidants and nanomaterials with new structures should be strengthened, and new synthesis techniques should be adopted to reduce prices. At the same time, we should strengthen the collaborative use of antioxidants, nanomaterials and
aromatic compounds to modify polymer materials in order to develop new radiation-resistant modification processes and polymer materials with better comprehensive performance.

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