

元素杂化阻燃高分子材料的研究进展

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摘 要 介绍了高分子材料的阻燃研究现状,概述了阻燃元素杂化技术、各元素阻燃机理及元素杂化协同阻燃高分子材料的研究进展,同时对阻燃剂的研究方向进行了展望。

关键词 高分子材料,杂化技术,阻燃元素,协同阻燃

Research progress in element hybrid flame-retardant polymer

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Abstract The research status of flame-retardant polymer was introduced. The progress of retardant element hybrid technology, the flame retardant mechanism of various elements and synergistic flame retardant polymer materials with hybrid elements were summarized, and the research direction of flame retardant was discussed.

Key words polymer, hybrid technology, flame-retardant element, synergistic flame retardant

高分子材料是科学技术和经济建设中的重要材料,由于机械强度高、质量轻和易于加工的优点,逐渐取代传统的无机材料、金属和木材^[1-2]。然而,高分子材料属于易燃材料,并且燃烧过程中有大量浓烟和有毒气体释放^[3-4]。因此,研究高分子材料的阻燃具有深远的意义。阻燃体系主要有磷、氮、硅和硼等单元素阻燃及元素杂化阻燃和无机填料阻燃。而阻燃剂则是通过共混、共聚、溶胶-凝胶和有机改性等方法添加到材料中以提高其阻燃效应^[5-6]。

1 阻燃元素的杂化

1.1 元素共混法

制备复合材料最简单最常用的方法是元素共混法。熔融共混和溶液共混是元素共混的 2 种主要方式。熔融共混是在粘流温度以上将聚合物与填料在混炼机混合从而制备聚合物共熔体。溶液共混是采用溶剂将填料溶解与聚合物混合均匀,然后加热蒸发除去溶剂即制得复合材料^[7]。共混法适用于多种形态的无机纳米粒子,而且共混时容易控制组分的浓度。然而相比其他杂化技术的不足在于有机或无机组分容易发生聚集,导致与基体材料相容性差^[8]。

1.2 元素共聚法

元素共聚法是以化学键键合的方式将阻燃化合物作为共聚单体引入高分子基体中,这种衔接方式可通过阻燃单体的结构对高分子链产生更加直接的影响,从而使高分子材料表现所需的性能^[9]。Liu 等^[10]合成了含磷三官能团反应型单体与环氧树脂共聚制备含磷阻燃环氧树脂。研究表明含磷基团阻燃剂的添加提高了环氧树脂的阻燃性以及物理性能。从数据分析表明,材料的热稳定性取决于其不同的交联密度。Wang 等^[11]采用 9,10-二氢-9-氧杂-10-磷菲-10-氧化物(DOPOM)和 1,3,5-异氰尿酸三缩水甘油酯(TGIC)合成磷、氮双元素单体 TGICP 阻燃环氧树脂。通过阻燃环氧树脂燃烧后的碳层形貌可以看出,添加阻燃剂的碳层更加致密光滑,这种碳层隔绝了可燃性气体与氧气的交换,从而提高了复合材料的热稳定性及阻燃性能。

1.3 溶胶-凝胶法

溶胶-凝胶法是以含活性组分的化合物为前驱体,在熔融状态下与原料混合均匀,并水解、缩合形成溶胶,溶胶经聚合形成凝胶后再干燥固化制得复合材料^[12-13]。Tsai 等^[14]以醚酐(ODPA)、对氨基苯基三甲氧基硅烷(APTS)和二氨基二苯基醚

基金项目:国家自然科学基金(51465036);甘肃省自然科学基金(1112RJZA019)

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(ODA)反应制备了甲氧基前驱体,再加入苯基三甲氧基硅烷(PTS)缩合,制备了具有网状结构的阻燃复合材料。

1.4 有机改性

无机粒子对聚合物力学性能的影响取决于在复合材料基体中的分散程度,对无机粒子进行表面有机改性,可提高与复合材料的相容性,从而改善对材料物理学性能的影响^[15-16]。功能化表面改性后的无机粒子可提高材料的抗老化性、热稳定性和阻燃性^[17]。

Cinausero等^[18]对纳米氧化铝粒子进行表面改性后,制得阻燃聚甲基丙烯酸甲酯(PMMA)。PMMA上的酯基与氧化铝(Al_2O_3)上的羟基发生反应形成羧酸酯,化学键的相互作用抵消了热分解前期氧化效应引起的聚合物链断裂,宏观上表现出聚合物材料的热稳定性有所提高。Ma等^[19]采用阻燃剂聚磷酸盐(PDSPB),以共价键的形式接枝到碳纳米管表面制备纳米复合材料(MWNT-PDSPB),并研究其阻燃性能。研究结果表明,MWNT-PDSPB的网状结构限制了复合材料高分子链的运动,提高了材料的阻燃性能。

1.5 微胶囊阻燃

微胶囊阻燃技术是以无机或有机物为壁材,阻燃剂为芯材,采用化学法、物理化学法和物理机械法等对阻燃剂进行包裹,从而形成微胶囊阻燃剂^[20]。微胶囊阻燃剂有以下这些优势^[21-23]:(1)将液态阻燃剂转变为固态,降低了阻燃剂的挥发性;(2)微胶囊壁材隔绝了阻燃剂与外界环境,提高了阻燃剂的耐久性;(3)微胶囊化的阻燃剂提高了与基体材料的相容性,降低了对材料力学性能的影响。

微胶囊阻燃剂解决了传统添加型阻燃剂与基体材料相容性差的问题。Liu等^[24]以聚磷酸铵(APP)为芯材,4,4-二氨基二苯醚(ODA)为壁材,制备微胶囊阻燃聚氨酯复合材料。研究表明,微胶囊化的APP能更有效提高聚氨酯的热稳定性、阻燃性能和极限氧指数。微胶囊阻燃是阻燃剂发展的方向之一。

2 单元素阻燃研究进展

2.1 磷元素阻燃

含磷化合物是阻燃环氧树脂最常用最有效的方法。磷系阻燃剂有2种:无机磷系阻燃剂和有机磷系阻燃剂。无机磷系主要有:红磷、磷酸盐和聚磷酸铵等;有机磷系主要是磷酸酯^[25]。磷系阻燃剂可在

凝聚相和气相同时发挥阻燃效应。气相中,磷化合物在高温条件下释放磷氧自由基,与促进燃烧的自由基结合,从而终止燃烧的链反应。在凝聚相中,磷催化促进聚合物脱水成炭,炭层覆盖在聚合物表面隔绝了氧气,阻止了自由基的逸出^[26-27]。

Qian等^[28]采用DOPOM和TGIC合成了含磷阻燃剂TGIC-DOPO,在高温分解时形成惰性片段促进了活性基团末端自由基的淬灭。热失重研究表明,阻燃剂提高了复合材料的残碳量及初始分解温度。极限氧指数和垂直燃烧试验表明TGIC-DOPO改善了环氧树脂的阻燃性。

2.2 氮元素阻燃

氮元素阻燃通常是以多聚磷酸铵,三聚氰胺盐等直接与基体材料复合。通过高温分解产生的难燃性含氮化合物稀释火焰附近燃烧气体的浓度起到阻燃效应。Zhao等^[29]合成了三聚氰胺盐阻燃聚乳酸,研究表明,含氮化合物可促进膨胀碳层的形成,这种膨胀碳层阻止了热传递和质量损失,还抑制了熔滴的形成。

2.3 硅元素阻燃

含硅化合物的阻燃效应发生在凝聚相。高温分解条件下,硅元素被氧化成氧化硅,而氧化硅表面能低,容易从基体材料中迁移到聚合物表面,形成具有抗氧化性的炭化层^[30]。Wu等^[31]合成了有机硅树脂阻燃聚氨酯泡沫材料。在高温条件下,有机硅聚合物转化成致密的氧化硅层,保护了泡沫材料内部可燃物的溢出,降低了热释放速率,提高了热稳定性。

聚硅氧烷与基体材料的相容性较差,单纯的硅元素很难达到理想的阻燃效应。硅元素与其他元素复配则能发挥更好的阻燃性能^[32]。Deh等^[33]研究表明,硅元素的引入提高了碳层的热稳定性增加了残碳质量,减少了所需的磷元素含量。

2.4 硼元素阻燃

硼系阻燃剂主要在凝聚相发挥阻燃效应,含硼化合物受热分解产生硼酸酐或硼酸(H_3BO_3)形成玻璃状的熔融物覆盖在材料表面,隔绝氧气和热量的传播,可以抑制浓烟和熔滴的产生^[34-35]。凝聚相不仅可以促进聚合物成炭,其本身也可在材料燃烧时生成类似陶瓷结构的多孔炭层,有利于隔热和阻止空气扩散进入材料内部^[36]。Çakmakç等^[37]研究表明,硼元素的加入改善了涂层的阻燃性能、热稳定性。 H_3BO_3 的羟基与聚合物材料间的相互作用也可提高涂层的弹性模量以及聚合物的抗拉强度。

2.5 铝、镁元素阻燃

铝、镁元素阻燃剂主要是由含羟基的氢氧化铝 $[\text{Al}(\text{OH})_3]$ 、氢氧化镁 $[\text{Mg}(\text{OH})_2]$ 等无机化合物组成^[38]。 $\text{Al}(\text{OH})_3$ 及 $\text{Mg}(\text{OH})_2$ 受热时键合在铝、镁上的羟基会分解生成水,在降低材料温度的同时稀释了可燃物质的浓度。 $\text{Al}(\text{OH})_3$ 及 $\text{Mg}(\text{OH})_2$ 脱水后生成的无机金属氧化物层覆盖在材料表面,起到很好的隔热效果,避免了基体材料在受热条件下的分解^[39-41]。

无机纳米粒子与聚合物材料的相容性和分散性较差^[42],对无机粒子进行超细化、有机改性可改善对材料力学性能的影响。Zhang 等^[43]合成了硼酸衍生物(3TT-3BA)与 $\text{Mg}(\text{OH})_2$ 的协同阻燃环氧树脂。3TT-3BA 与 $\text{Mg}(\text{OH})_2$ 的复配提高了材料的热稳定性和阻燃性能。3TT-3BA 的加入改善了 $\text{Mg}(\text{OH})_2$ 对复合材料力学性能的影响。

3 元素杂化协同阻燃体系

3.1 磷-氮阻燃体系

磷-氮阻燃体系是研究最多的元素杂化协同阻燃体系^[44]。磷元素在凝聚相阻燃中可形成稳定的含磷碳层。氮元素在高温下生成无毒的难燃性气体,可以稀释可燃气体的浓度^[45-46]。磷、氮元素协同形成发泡的多孔膨胀碳层,此层可阻燃可燃性气体进入气相达到隔热、隔氧的作用^[47]。膨胀型阻燃剂需要酸源(脱水剂)、碳源和气源才能形成膨胀碳层。Wang 等^[48]合成了磷-氮共聚物阻燃环氧树脂,磷-氮协同阻燃比单一元素阻燃环氧树脂的热稳定性高。

3.2 磷-硅阻燃体系

磷-硅协同阻燃复合材料在高温条件下,含磷基团分解生成的偏磷酸脱去基体材料中的水形成含磷碳层^[49]。由于硅元素表面能较低,容易迁移至材料表面形成含硅保护层,提高了材料表面碳层的热稳定性,抑制了含磷碳层高温条件下被氧化^[50]。Hamciuc 等^[51]研究表明,硅元素的加入可提高聚合物的初始分解温度和玻璃化转变温度,增强复合材料的热稳定性。磷、硅元素的协同效应避免了材料表面碳层的氧化。

3.3 氮-硅阻燃体系

阻燃复合材料时,硅元素分解生成氧化物碳层,氮元素生成难燃性的含氮气体,则磷、硅协同阻燃在凝聚相和气相发挥阻燃效应。Hsiue 等^[52]以含硅固化剂为硅源添加至环氧树脂制备阻燃复合材

料。硅元素的引入提高了材料的分解温度和残留物质量。

4 发展趋势

从上述研究成果可以看出,高分子材料的阻燃以多元素杂化协同阻燃为主要研究趋势,由于传统阻燃剂对基体材料力学性能的影响较大,将阻燃剂有机改性和纳米粒子化,是研究阻燃剂与基体材料相容性差、降低力学性能问题的重要方向。随着对环境保护的重视,发烟量低、毒性小和环境友好型阻燃剂的开发成为必然趋势。本征阻燃剂合成工艺复杂、生产成本低,不利于工业化生产,而低成本、工艺简单的阻燃剂是阻燃市场的发展方向。随着材料领域的发展,功能化阻燃剂是未来阻燃复合材料研究的新趋势。

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收稿日期: 2017-11-24

修稿日期: 2019-02-21

麻省理工: 发现内含子对酵母生长的调节机制

近日, 美国麻省理工学院研究人员针对内含子对酿酒酵母生长的调节作用进行了研究。通过对已知内含子进行删除, 研究内含子缺失对酵母生长的影响, 为真核单细胞生物内含子的生物学功能研究提供新视角。

内含子是真核生物体内普遍存在的非编码 RNA, 它在新生 mRNA 中被剪接去除以产生功能性蛋白质, 但其生物学功能至今仍未被阐释清楚。

研究人员对酿酒酵母的 34 个内含子进行了删除, 发现虽然线性 RNA 在饱和生长条件或其他应激条件下进

行积累, 但在对数期生长期中迅速降解, 从而长期抑制生长信号 TORC1。这些内含子的删除对饱和和生长条件下的酵母是不利的, 并且在 TORC1 抑制剂雷帕霉素作用下, 酵母生长速率异常升高。回补相应的天然或人工内含子可以有效抑制雷帕霉素导致的异常作用。此外, 内含子的稳定与剪接体的组分相关, 较近的套索分支点和 3' 端剪接位点间距是内含子稳定的必要条件。该研究揭示了内含子在酿酒酵母 TOR 生长信号传导网络内的作用及剪接体内含子的生物学功能。(新型)