

水玻璃改性聚氨酯发泡灌浆材料的制备与性能研究

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摘要:针对引调水隧洞施工与运营过程中突涌水及渗漏等灾害治理难度大的问题,以多苯基多亚甲基多异氰酸酯(PAPI)、水玻璃(WG)和聚醚多元醇等为原料,通过有机/无机杂化的方式制备出水玻璃改性油性聚氨酯(PU/WG)发泡灌浆材料。宏观性能结果表明:水玻璃最优掺量为20%,此时改性聚氨酯密度为 1.102 g/cm^3 ,发泡率可达2684%,7d抗压强度可达38.9 MPa。扫描电子显微镜(SEM)测试微观分析表明:随着水玻璃掺量的增加,光滑平整的聚氨酯表面出现球形泡孔后逐渐被不定形无机相覆盖,最终呈现为疏松多孔结构。热重分析(TGA)表明:改性聚氨酯热稳定性明显优于纯聚氨酯。研究成果可为改性油性聚氨酯灌浆材料的性能提升和体系优化提供数据支撑。

关键词:引水隧洞;突涌水;聚氨酯;水玻璃改性;发泡灌浆材料;扫描电子显微镜(SEM);热重分析(TGA);抗压强度
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Preparation and Properties of Polyurethane Foam Grouting Material Modified by Waterglass

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Abstract: [Objective] Grouting has been recognized as an effective solution for water inrush treatment in underground engineering. Organic grouting materials such as polyurethane (PU) are strongly adhesive but expensive, whereas inorganic grouting materials such as waterglass (WG) are cheap but have low elasticity. To improve the performance of polyurethane grouting materials, we investigated the influence of waterglass content on the macroscopic and microscopic properties of polyurethane. [Methods] Polyphenyl polymethylene polyisocyanate (PAPI), waterglass, and polyether polyol were used as the basic raw materials, and the inorganic/organic hybrid method was adopted to prepare polyurethane foam grouting material modified by waterglass. The influence of waterglass content by weight (0, 20%, 40%, 60%, 80%) on the foaming rate, density, and compressive strength of the modified polyurethane was investigated. A fitting relationship between polyurethane density and elastic modulus was established. Scanning electron microscopy (SEM) and thermogravimetric analysis (TGA) were used to measure the influence of waterglass content on the cross-sectional morphology and thermal stability of materials. [Results] (1) Macroscopic property results showed that, with increasing waterglass content, both the foaming rate and compressive strength first increased and then decreased. When the waterglass content was 20%, the foaming rate and compressive strength reached maximum. Under this condition, the foaming rate was 2684%, and the compressive strength at 7 d was

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38.9 MPa. In addition, the heat released by the reaction increased the instability of the cell structure and even caused cell collapse, resulting in decreases in foaming rate and compressive strength. Notably, when the density changed from 1.102 g/cm³ to 0.959 g/cm³, the compressive strength of materials decreased by approximately 70%. Based on the experimental results, the fitting relationship between the density and elastic modulus of the modified polyurethane was obtained as $E \propto \rho^{1.8}$. (2) The results of scanning electron microscopy showed that, with increasing waterglass content, the smooth polyurethane surface was gradually covered by an amorphous inorganic phase surrounding the spherical cells. The structure of the materials eventually became loose and porous. (3) TGA results showed that the thermal stability of the modified polyurethane was better than that of pure polyurethane. The TG curve showed that when $T < 80$ °C, the thermal mass loss of the materials was only about 5%, indicating that the initial decomposition temperature of the materials was about 80 °C. When $T > 600$ °C, the curve became stable, meaning that the modified polyurethane with waterglass content of 0, 20%, 40%, 60%, and 80% lost approximately 84%, 77%, 72%, 65%, and 53% of their mass, respectively. The curves then tended to stabilize, indicating that the incorporation of waterglass could enhance the thermal stability of polyurethane. (4) DTG (Differential Thermogravimetry) curve showed that the mass loss rate of polyurethane was significantly higher than that of the modified polyurethane. This was attributed to the fact that the inorganic components precipitated in the reaction system were encapsulated on the surface of polyurethane matrix. The presence of Si-O bonds increased the intermolecular forces, resulting in the need for more energy to cause thermal decomposition of the materials. Therefore, the addition of waterglass effectively reduced the thermal decomposition rate of polyurethane. [**Conclusion**] Polyurethane/waterglass (PU/WG) is an organic-inorganic hybrid material that has the advantages of both polyurethane and waterglass. Analysis of the relationship between waterglass content and the macroscopic and microscopic properties of the modified polyurethane shows that the addition of waterglass helps increase the foaming rate, compressive strength, and thermal stability of polyurethane. These findings offer data support for the performance improvement and system optimization of the modified oil-soluble polyurethane grouting materials.

Key words: headrace tunnel; water inrush; polyurethane; waterglass modification; foam grouting materials; scanning electron microscopy (SEM); thermogravimetric analysis (TGA); compressive strength

0 引 言

我国水资源分布和生产布局存在着明显的区域矛盾,加快构建国家水网,建设大型的跨流域调水系统是优化基础设施布局,实现国家水资源优化配置的重要举措^[1-2]。

深埋长隧洞作为大型调水工程的重要载体,建设过程中不可避免需穿越深山峡谷地带,如何处理施工与运营过程中面临的高压大流量突涌水及渗漏问题成为决定工程成败的关键因素^[3-7]。灌浆作为隧洞水灾害治理的主要措施,在突涌水灾害控制、渗漏水灾害处理中效果显著。在突涌水灾害灌浆处理过程中,材料性能优劣与价格高低直接影响灌浆处理效果和工程成本。通常灌浆材料主要包括水泥基、环氧树脂和聚氨酯等。水泥基灌浆材料成本低,但强度上升慢且基本无发泡效果;环氧树脂灌浆材料强度高,但价格偏高且有水环境下固化效果相对较差;聚氨酯灌浆材料对于治理隧洞施工与运营过

程中的渗漏水、突涌水灾害具有明显优势^[8]。聚氨酯主要原料多元醇类型丰富,通过调整多元醇等原材料的种类和用量,可获得性能各异的聚氨酯灌浆材料^[9-11]。

通常情况下,隧洞灌浆堵水工程往往材料耗量较大,采用纯聚氨酯浆材成本较高,同时深部岩体中的高地温环境对于材料的热稳定性亦提出较高要求。因此,开展堵水填充型灌浆材料的研究,研发低成本、发泡率和力学强度等可调可控的灌浆材料处理隧洞施工中遭遇的突涌水、运营期的渗漏水,实现对水灾害的防治与处理具有重要工程价值。

为开发出低成本、综合性能优良的聚氨酯灌浆材料,一方面可针对纯聚氨酯进行配方优化,另一方面开发出性能优良的改性聚氨酯。近年来,科研工作者主要采用无机材料对聚氨酯开展改性研究,以获得低成本、性能优良的改性聚氨酯灌浆材料。如不同学者采用预聚体法和原位聚合法在水溶性聚氨酯制备过程中掺入纳米 SiO₂ 以期提高材料压缩强度,发现改性水溶性聚氨酯强度最大可提升 50% 左

右,可纳米 SiO₂的参加在一定程度上影响材料凝胶时间和发泡率等性能^[12-13]。当前硅酸盐改性油性聚氨酯(Polyurethane Modified by Waterglass, PU/WG)灌浆材料亦取得一定的研究成果^[14-15]。如 He 等^[16]采用硅烷偶联剂对水玻璃(Waterglass, WG)进行预处理,制备出一种改性油性聚氨酯灌浆材料,试验结果表明偶联剂在反应体系中同时发挥偶联剂和表面活性剂的效果,预处理后的水玻璃均匀分散于聚氨酯基体中,改性聚氨酯抗压强度同比提高11.65%以上。

此外,不同学者针对催化剂的种类和用量对改性油性聚氨酯力学性能的影响也有一定的研究报道。如张茜等^[17]对比研究了单一催化和复合催化对改性聚氨酯灌浆材料压缩强度的影响,研究表明复合催化作用下材料综合性能相对较优,压缩强度可达 66.1 MPa。柏广峰^[18]亦开展了复合催化作用下改性聚氨酯力学强度对比研究,研究表明在复合催化作用相近的情况下,添加三聚催化剂有望在反应体系中生成异氰尿酸酯环,从而提高材料力学性能。目前,对硅酸盐改性油性聚氨酯在不同影响因素下如催化剂、偶联剂等对力学强度的影响已有大量研究,可对不同水玻璃掺量下材料密度、热稳定性、发泡率和抗压强度及其关联性研究不够充分。

本文基于课题组前期研究^[19],采用低廉的水玻璃对油性聚氨酯开展改性研究,制备出堵水发泡型改性聚氨酯灌浆材料,进一步通过扫描电子显微镜(Scanning Electron Microscope, SEM)和热重分析(Thermogravimetry Analysis, TGA)对试样开展表征,探究了水玻璃掺量对材料密度、发泡率、抗压强度、热稳定性和微观形貌的影响,为后续改性油性聚氨酯性能优化提供科学依据。

1 试验材料与设计

1.1 原材料

试验主要原料有多苯基多亚甲基多异氰酸酯(Polyphenyl Polymethylene Polyisocyanate, PAPI)(牌号 PM 2010),万华化学集团股份有限公司生产;多元醇(牌号 DL-2000D),济南荣正化工有限公司生产;水玻璃(波美度 30~50,模数 2.6~3.0),青岛东岳泡花碱有限公司生产;N,N-二甲基苄胺(N,N-Dimethylbenzylamine, BDMA),广州市润宏化工有限公司生产;有机硅表面活性剂(牌号 BL-8468),广州市润宏化工有限公司生产;增塑剂二

甘醇二苯甲酸酯(Diethylene glycol dibenzoate, DEDB)、己二酸二辛酯(Dioctyl adipate, DOA),莱阳市康德化工有限公司生产。

1.2 材料制备

(1)甲组分的制备:将一定比例的 PAPI、多元醇和催化剂等加入塑料杯中,搅拌至均匀备用。

(2)乙组分的制备:以甲组分质量作为参考,将不同质量百分比(0%、20%、40%、60%、80%)的水玻璃在电动搅拌器 300 r/min 条件下搅拌 3 min 备用。

(3)复合材料的制备:将甲组分和乙组分混合,以 300 r/min 的速度均匀搅拌 2 min。硅酸盐改性聚氨酯发泡灌浆材料各成分用量及加入比例见表 1。

表 1 PU/WG 灌浆材料各成分用量及加入比例

Table 1 Components of PU/WG grouting materials

甲组分/g						乙组分/%
PAPI	DL-2000D	BDMA	BL-8468	DEDB	DOA	水玻璃
33	8	2	5	8	8	0~80

1.3 测试方法

发泡率和抗压强度测试参照《聚氨酯灌浆材料》(JC/T 2041—2020)^[20]规定的方法进行。改性聚氨酯抗压强度试验采用尺寸为 50 mm×50 mm(直径×长度)的圆柱体试件,每组制作 3 个试件。

SEM 测试采用荷兰 FEI 公司生产的 nova 场发射扫描电子显微镜进行试验,从抗压强度试样破坏面处收集样品,获得图像并开展对比分析;TGA 分析采用铂金-埃尔默仪器(上海)有限公司的 TG/DTA6300 型热重分析仪进行测试。

2 试验结果分析

2.1 水玻璃掺量对试样发泡率影响分析

在突涌水封堵过程中,灌浆材料具有优良的发泡率有助于快速填充渗漏水通道,同时反向压密破碎围岩。不同掺量(0%、20%、40%、60%、80%)水玻璃制备出的改性聚氨酯发泡率试验结果见图 1。

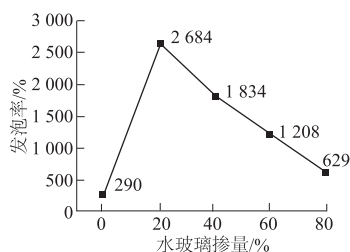


图 1 水玻璃掺量对试样发泡率的影响

Fig.1 Influence of waterglass content on foaming rate of samples

由图 1 可知:随水玻璃掺量的增加,发泡率先升高后降低。当水玻璃掺量为 20%时,发泡率达到最

大值 2 684%。上述情况一方面可归因于当体系内水玻璃过量, —NCO 与水发生反应产生 CO₂ 致使材料发泡, 由此形成的泡孔结构脆性偏高, 加之反应放热致使泡孔结构不稳定程度增加甚至产生塌孔; 另一方面, 过量的水玻璃在体系中逐渐析出, 无机成分增多进一步增大了材料脆性, 发泡率随之降低。

2.2 水玻璃掺量对试样密度和强度影响分析

不同水玻璃掺量下试样抗压强度和密度变化曲线见图 2。

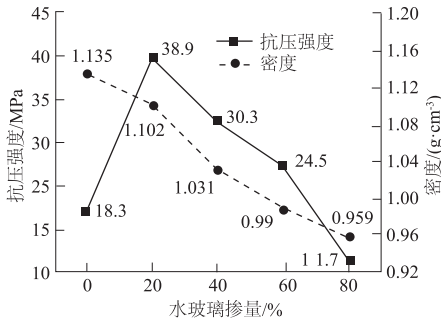


图 2 水玻璃掺量对试样密度和抗压强度的影响

Fig.2 Influence of waterglass content on density and compressive strength of samples

由图 2 可知: 随水玻璃掺量的增加, 试样抗压强度先升高后降低。当水玻璃掺量为 20% 时, 抗压强度达到最大值 38.9 MPa; 继续掺入水玻璃, 试样抗压强度逐渐降低。上述情况归因于当水玻璃掺量较少时, 其水解生成的活性硅醇与 —NCO 发生反应, 在聚氨酯链段中引入 Si—O 键, 形成无机-有机互穿网络结构, 促使材料强度升高。可掺入过量水玻璃, 由于无机相与有机相的界面差异性, 多余的水玻璃并未与聚氨酯完全结合以提高强度, 反之在体系中引入过量的脆硬性无机 Si—O 键致使材料脆性增加。值得注意的是: 当试样密度从 1.102 g/cm³ 变化为 0.959 g/cm³ 时, 材料抗压强度降低近 70%, 故针对材料密度与强度相关性开展研究, 试样密度对抗压模量的影响见图 3, 初步得出改性发泡聚氨酯密度 ρ 与弹性模量 E 的拟合关系式为 $E \propto \rho^{1.8}$ 。

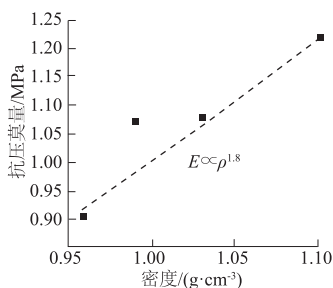


图 3 试样密度对抗压模量的影响

Fig.3 Influence of density on compressive modulus of samples

2.3 SEM 分析

不同水玻璃掺量下试样微观形貌见图 4。由图 4(a) 可知, 纯聚氨酯表面相对光滑平整, 未出现孔洞和微裂隙等; 由图 4(b) 可知, 当水玻璃掺量为 20% 时, 聚氨酯基体表面出现球形泡孔; 继续掺入水玻璃, 由图 4(c) 可知, 当水玻璃掺量为 40% 时, 球形泡孔表面开始出现无机相, 材料表面光滑程度明显降低, 呈现疏松多孔趋势; 由图 4(d) 可知, 当水玻璃掺量为 60% 时, 材料表面的无机相逐渐增多且形状无规则, 这将在一定程度上出现局部应力集中问题; 由图 4(e) 可知, 当水玻璃掺量增大到 80% 时, 球形泡孔表面被不定形无机相覆盖, 材料整体逐渐呈现为疏松多孔结构, 导致材料力学强度降低。微观结构上无序的相分布导致应力集中, 宏观性能下降。SEM 分析结果表明: 随水玻璃掺量的变化, 改性聚氨酯形貌演变规律与宏观性能变化趋势一致。

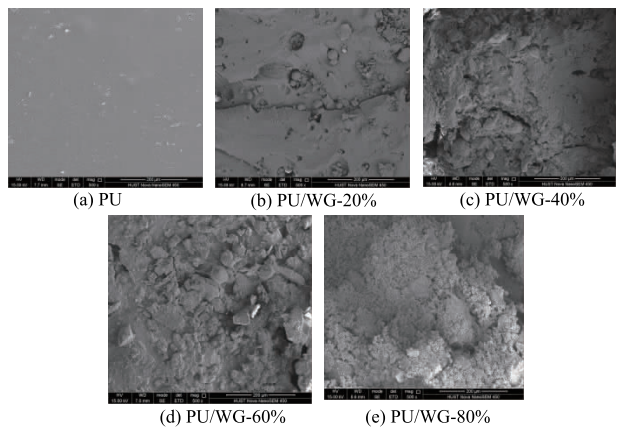


图 4 不同掺量的水玻璃改性聚氨酯 SEM 图像

Fig.4 SEM images of polyurethane modified with different waterglass contents

2.4 热重分析

TGA 可精确测量样品在控温过程中的质量变化过程, 从而得到试样分解时的温度区间范围, 分析样品的热稳定性。在此基础上, 对 TG 曲线进行微分处理得到热重微分曲线 (Differential Thermogravimetry, DTG), 得到试样在温度变化过程中质量变化速率和最大变化速率下对应的特征温度等。不同水玻璃掺量下试样 TG 曲线和 DTG 曲线见图 5。

由图 5(a) 可知: 当温度 $T < 80$ °C 时, 试样热质量损失相对较低, 只有 5% 左右的少量失重, 即材料的初始分解温度在 80 °C 左右; 当温度 $T > 600$ °C 时, TG 曲线呈现水平状态, 即水玻璃掺量为 0%、20%、40%、60%、80% 的试样, 质量分别损失 84%、77%、72%、65% 和 53% 左右后趋于稳定, 说明掺入水玻璃可提高聚氨酯的热稳定性。

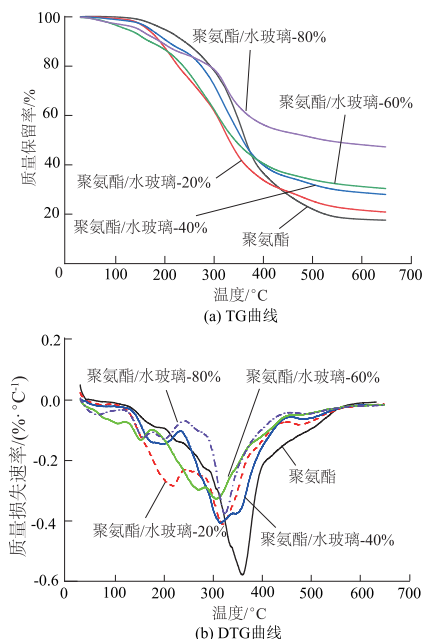


图5 聚氨酯及改性聚氨酯的TG及DTG曲线

Fig.5 TG and DTG curves of polyurethane and modified polyurethane

由图5(b)可知:DTG曲线呈现3个峰,对应材料热分解的3个阶段。第一阶段80~110℃与水分的挥发和CO₂的释放有关;第二阶段110~250℃与氨基甲酸酯、脲键和Si-O键的热分解有关;第三阶段250~600℃与材料中多元醇、硅醇的热分解有关,本阶段聚氨酯质量损失速率明显高于改性聚氨酯,归因于反应体系中析出的无机成分包裹在聚氨酯表面,Si-O键的存在增加了分子间作用力,导致需要更多的能量才能使改性聚氨酯产生热分解,硅醇产生热分解后聚氨酯才发生热分解,即掺入水玻璃有效降低了聚氨酯热分解速率。

3 结论

本文研究了水玻璃掺量对改性聚氨酯密度、发泡率和抗压强度的影响。在此基础上,开展不同水玻璃掺量下材料形貌和热重分析,得到结论如下:

(1)水玻璃最优掺量为20%,此时改性聚氨酯密度为1.102 g/cm³,发泡率达到2684%,抗压强度达到38.9 MPa,改性聚氨酯密度与弹性模量的拟合关系式为 $E \propto \rho^{1.8}$ 。

(2)对比不同水玻璃掺量下改性聚氨酯的微观形貌,发现水玻璃掺量增加将加剧泡孔结构不规则程度,球形泡孔表面被不定形无机相覆盖,材料整体逐渐呈现出疏松多孔结构。

(3)改性聚氨酯初始分解温度在80℃左右,并在600℃左右质量趋于稳定。不同水玻璃掺量下改

性聚氨酯的TG和DTG曲线表明掺入水玻璃提高了聚氨酯的热稳定性,有效降低其热分解速率。

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