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Overlooked Impact of Interchain H-Bonding between Cross-Links on the Mechanical Properties of Thermoset Polyurethane Elastomers

Zhishuai Geng, Aimin Pang, Tengfei Ding, Xiaoyan Guo, Rongjie Yang, Yunjun Luo, and Jinxian Zhai*



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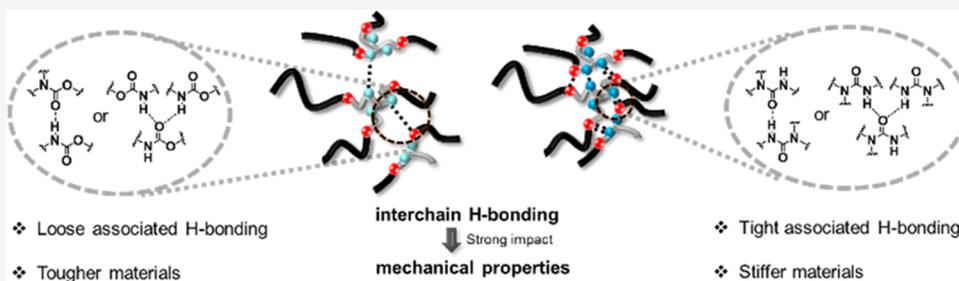
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ABSTRACT: Thermoset polyurethane elastomers made from poly(3,3-bis(azidomethyl) oxetane with tetrahydrofuran) and various multifunctional isocyanate cross-linkers were compared to uncover a new mechanism of modulating the mechanical properties. Extra hydrogen bonding motifs, such as urethane or urea, were built in the cross-linkers and were proved to essentially determine the stiffness and toughness of the elastomers, while the covalent cross-linking densities of both networks were controlled strictly at the same level. The impact of interchain H-bonding on the mechanical properties of the polyurethane thermoset was unprecedentedly emphasized and supported by evidence from Fourier-transform infrared spectroscopy (FTIR), dynamic mechanical analysis (DMA), and low-field nuclear magnetic resonance (LFNMR).

INTRODUCTION

Polyurethane elastomers are an important kind of visco-elastic material, with a relatively low elastic modulus at a certain temperature scope and large reversible deformability.^{1,2} Thermoplastic polyurethane elastomers (TPE) are made up of hard segments (HS) and flexible prepolymer soft segments (SS). Varying the structure and content of HS by using various isocyanates and chain extenders would allow for adjustment of hydrogen bonding (H-bonding) strength, microphase separation structure and morphology, and physical cross-linking degree, imparting TPE excellent mechanical properties³ (Figure 1a). Nevertheless, the reversibility of H-bonding, which gives TPE the advantage of being able to be reprocessed, causes segments' transient fluidity at elevated temperature or upon deformation, raising creep, hysteresis, and lack of long-term mechanical performance stability. A series of multiple H-bonding moieties, such as ureidopyrimidone with high affinity and directionality, are introduced into TPE to achieve high mechanical performance.^{4–8} However, this novel design usually leads to changes in bulk properties and involves nontrivial monomer synthesis.

To enhance the mechanical performance while retaining the reprocessibility, polyurethane-based vitrimers or covalent adaptable networks have attracted extensive attention recently.^{9–11} Many dynamic covalent bonds have been introduced into the polyurethane thermoset to endow the network with malleability under certain stimuli, such as

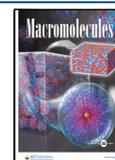
disulfide,^{12,13} furan-maleimide,¹⁴ acetal,¹⁴ and oxime.^{15,16} Besides, the transcarbomoylation exchange reaction is also taken advantage of to facilitate the reshuffling of polyurethane chains at elevated temperature or with the assistance of a catalyst.^{9,17,18} The novel dynamic covalent polyurethane network is still in the exploring state and requires further study before being put into wider applications.

Conventional covalently cross-linked thermoset polyurethane elastomers, based on SS strands and cross-links (usually made from isocyanates and low-molecular-weight polyols, thus defined as HS in thermosets), are handily prepared and are still predominant in many special application scenarios because they could effectively retain the network topology features, dimensional stability, creep, and chemical resistance.¹⁹ However, the systematic analysis of the mechanical properties of polyurethane thermosets is not as rich as that of TPE. The majority of the strategies on tuning their mechanical properties have been focused on adjusting the cross-linking density, SS prepolymer modification and blending, mixing of cross-linkers

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Neighboring Group Participation in Ionic Covalent Adaptable Networks

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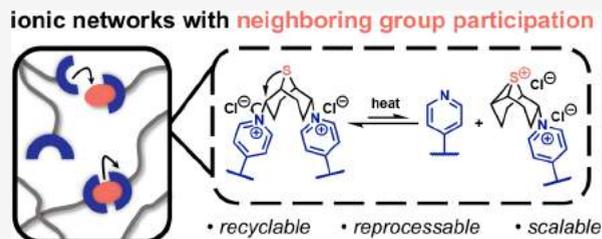


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ABSTRACT: Covalent adaptable networks (CANs) typically require external catalysts for efficient cross-linker exchange, which can limit network reprocessability due to catalyst leaching and degradation. In this study, catalysts were avoided by using a bicyclo[3.3.1]nonane bis-alkyl halide cross-linker with sulfur-atom neighboring group participation (NGP) to increase the rate of bond exchange. Stress relaxation analyses demonstrate that the resultant pyridine-based network has an Arrhenius dependence on viscous flow at elevated temperatures (130–170 °C), which arises from S_N1 transalkylation exchange. This thermally mediated cross-link interchange and associated flow behavior enabled reprocessing of the ionic networks over multiple damage and repair cycles. Additionally, these NGP-based CANs are chemically recyclable, allowing for recovery of the pyridyl-based polymer starting material, which comprises >90 wt % of the parent network. The dual thermal and chemical recycling potential of this catalyst-free CAN platform addresses key criteria for designing thermosets with extended lifecycles.



INTRODUCTION

Covalently cross-linked polymer networks, better known as thermosets, have become ubiquitous materials owing to their solvent resistance, mechanical strength, as well as thermal and dimensional stability.^{1,2} While covalent cross-linking imbues thermosets with desirable properties, it comes at the expense of recyclability and sustainability. To combat the environmental challenges posed by traditional thermoset end-of-life fates (landfilling and incineration), the pursuit of reprocessable and chemically recyclable alternatives holds significant promise for future polymer science research targets.^{3–5}

To develop thermoset plastics and rubbers with more circular lifecycles, a class of networks composed of cross-links that exchange in response to an external stimulus has been developed and termed covalent adaptable networks (CANs).⁶ These CANs combine two long-established phenomena: dynamic covalent chemistry and chemical stress relaxation.^{7–9} More than 70 years ago, Tobolsky and Stern demonstrated that polysulfide rubbers underwent stress relaxation at elevated temperatures upon application of a step strain.⁹ Shortly thereafter, this stress decay process, dubbed chemorheology, was observed in silicone and polyurethane elastomers.^{10–12} Such chemical stress relaxation arises from dynamic covalent bonds (e.g., disulfides) within the network, which undergo reversible exchange when exposed to elevated temperatures and typically, the presence of a catalyst.^{10,11}

As the field of CANs has matured and the range of cross-linker exchange chemistries expanded,¹³ addressing common CAN limitations, such as the need for external catalysts, has

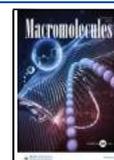
become a major research focus.^{14,15} For the purposes of this study, external catalysts are categorized as organic or inorganic molecules that are (1) not covalently bound to the network and (2) facilitate the thermal activation of dynamic covalent bonds. Although these catalytic additives are beneficial for lowering the energetic barrier for cross-linker exchange, they can promote degradation pathways within the surrounding polymer matrix at the high temperatures often needed to induce macroscopic flow in dynamic networks. Additionally, external catalysts can leach from CANs when exposed to solvents and have their own intrinsic thermal degradation profiles, which must be considered in conjunction with those of the bulk network. As a result, catalyst loss via degradation and/or leaching converts cross-links from dynamic to permanent, which impedes recyclability goals.

Recently, neighboring group participation (NGP) has been implemented with transesterification-based CANs as a strategy to replace external catalysts.^{16–18} Formally, NGP occurs when an atom or a functional group (e.g., π -bond) intramolecularly forms a bond (or partial bond) to the reaction site, stabilizing the resulting transition state or intermediate.¹⁹ When NGP imparts stabilization in the rate-determining step of the

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Review

Strategy for Constructing Phosphorus-Based Flame-Retarded Polyurethane Elastomers for Advanced Performance in Long-Term

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Abstract: Polyurethane elastomer (PUE), which is widely used in coatings for construction, transportation, electronics, aerospace, and other fields, has excellent physical properties. However, polyurethane elastomers are flammable, which limits their daily use, so the flame retardancy of polyurethane elastomers is very important. Reactive flame retardants have the advantages of little influence on the physical properties of polymers and low tendency to migrate out. Due to the remarkable needs of non-halogenated flame retardants, phosphorus flame retardant has gradually stood out as the main alternative. In this review, we focus on the fire safety of PUE and provide a detailed overview of the current molecular design and mechanisms of reactive phosphorus-containing, as well as P-N synergistic, flame retardants in PUE. From the structural characteristics, several basic aspects of PUE are overviewed, including thermal performance, combustion performance, and mechanical properties. In addition, the perspectives on the future advancement of phosphorus-containing flame-retarded polyurethane elastomers (PUE) are also discussed. Based on the past research, this study provides prospects for the application of flame-retarded PUE in the fields of self-healing materials, bio-based materials, wearable electronic devices, and solid-state electrolytes.

Keywords: polyurethane elastomer; phosphorus flame retardant; reactive flame retardant; fire protection



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1. Introduction to Polyurethane Elastomers

Polyurethane elastomer (PUE) is a typical block polymer with alternately arranged soft and hard segments [1,2]. Isocyanates and small molecular chain extenders constitute the hard segment, and the soft segment is composed of polyols (shown in Figure 1). Compounds containing two or more -OH groups with a molecular weight of more than 500 g/mol are defined as polyols, while those smaller molecules with hydroxyl groups and/or amines are regarded as chain extenders.

The abundant hydrogen bonds physically cross-link the linear chains to form network structures, resulting in microscopic phase separation between soft and hard segments [3]. The mobility of the soft segment makes the PUE elastic, while the hard segment hinders the rotation of the molecular chain, giving the PUE hardness and mechanical strength [4]. The unique micro-phase structure of PUE endows it with wear resistance, toughness, and good processability of thermoplastics [5]. The combination of “tough and strong” greatly expands the application of PUE.



Dual nucleation sites induced by ZIF-67 towards mismatch of polyphosphazene hollow sub-micron polyhedrons and nanospheres in flame retardant epoxy matrix

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ABSTRACT

Polyphosphazene has drawn an abundance of attention as a flame retardant due to its eco-friendliness and phosphorus-nitrogen synergistic effect. ZIF-67 and cobalt compounds derived from cobalt ions released by ZIF-67 were employed as dual templates for loading polyphosphazene. We skillfully designed a polyphosphazene hollow hybrid material via the polymerization of phosphazene accompanied by the etching of ZIF-67 and cobalt compounds by generated HCl. The hybrid material (CPPHS) was composed of cobalt-doped polyphosphazene hollow sub-micron polyhedrons (CP) and polyphosphazene hollow nanospheres (PHNS) with different dimensions and magnitudes of sizes. After adding 2.0 wt% CPPHS, the limiting oxygen index of epoxy resin increased to 27.6%, while the peak heat release rate and total smoke production decreased by 40.1% and 38.8%, respectively.

The formation of a continuous and strongly extended dense char layer in the EP composites was fostered by the synergistic effect of Co-P-N flame retardant components, significantly decreasing the heat flow and combustible gas exchange rate and effectively hindering the combustion chain reaction. Furthermore, the unique dimensional mismatch structure of CPPHS enhanced its dispersion in the EP matrix, enabling the EP to have superior mechanical properties that match realistic application requirements, consequently expanding the variety of applications for fire-resistant EP composites.

1. Introduction

Epoxy resin (EP) is a common and basic thermosetting resin that is widely used in construction, transportation, aerospace, and electronics owing to its remarkable chemical stability, strong bonding strength, low shrinkage, ease of processing, and cost-effectiveness [1–3]. However, like most polymer materials, EP has two distinct drawbacks: flammability and toxicity of smoke. Additionally, the high cross-link density of EP leads to poor toughness and low fracture energy, restricting its development in the field of advanced materials [4–6]. Thus, improving the flame retardancy, smoke suppression, and mechanical qualities of EP is of great importance in engineering [7]. The combination of phosphorus and nitrogen-based flame retardants has shown great potential in enhancing flame retardancy through heat insulation, smoke suppression, and low toxicity. Polyphosphazene is a type of organic–inorganic

hybrid material with a unique nitrogen-phosphorus structural unit. Owing to the synergistic effect between phosphorus and nitrogen, polyphosphazene is endowed with excellent thermal stability and flame retardancy, which is also key to its research as a flame retardant [8]. The synthesis route of linear polyphosphazenes is strictly controlled and the yield is limited. On the contrary, cyclomatrix polyphosphazenes are synthesized by a simpler method, and the yield is as high as 90%. By virtue of the highly cross-linked rigid structures, as well as strong molecular designability, cyclomatrix polyphosphazenes have become a major focus of research in the field of phosphorus-based flame retardants [9,10]. Yang et al. [11] prepared a novel polyphosphazene (PBFA) containing an active amine group in order to improve the fire safety of EP composites. When the addition amount was 9.0 wt%, the peak heat release rate (pHRR) and total smoke production (TSP) of EP/PBFA composites decreased by 46.7% and 48.0%, respectively. Lou et al.

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REVIEW

Click chemistry strategies for the accelerated synthesis of functional macromolecules

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Abstract

Click chemistry is one of the most powerful strategies for constructing polymeric soft materials with precise control over architecture and functionality. In this review, we provide a comprehensive summary of the state-of-the-art for synthesizing functional polymers and their expanding range of applications. The synthetic and mechanistic aspects are discussed for key reactions that fulfill “click” requirements and their applications in construction of macromolecules with linear, branched, and other complex architectures are described.

KEYWORDS

click chemistry, macromolecules, synthesis, polymerization, postpolymerization modification

1 | INTRODUCTION

There is an increasing necessity for polymeric materials with precise control over architecture, functionality, and reactivity in a range of industrial and academic settings. To fulfill these requirements, researchers have sought to prepare and functionalize polymer systems via simple and high-yielding chemistry. This is in accord with the “click” chemistry philosophy initially described by Sharpless and coworkers in 2001—modularity, mild reaction conditions, and quantitative yields.¹ Following this inspiration, polymer science has witnessed a broad adaption of click chemistries to afford macromolecular materials tailored for various applications. The scope of click

chemistry has also broadened as additional reactions are developed. Combined with controlled polymerization techniques, these efficient transformations have enabled complex architectures to be easily obtained.

This review is divided into three sections that highlight applications of a wide range of click chemistries for polymer synthesis and functionalization. We begin with an introduction of current reactions that fit the philosophy of click chemistry, including the traditional click reactions such as azide-alkyne cycloaddition (AAC), thiol-ene, and Diels–Alder reactions. In addition, we focus on the discussion of emerging and underdeveloped click reactions, such as oxime ligation, triazolinedione (TAD)-based chemistry, and Sulfur(VI)-Fluoride Exchange (SuFEx), particularly in terms of mechanism, substrate scope, and orthogonality. The following section describes the use of these click chemistries for

Zhishuai Geng, Jaeman J. Shin, and Yumeng Xi authors contributed equally to this work.

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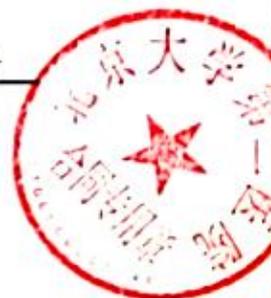
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第六条: 违约责任

1. 甲方未按照合同约定向乙方提供技术资料 and 协作事项的, 乙方有权要求甲方在合理期限内提供, 由此延误的技术服务期限予以顺延。

2. 甲方无正当理由未按照合同约定期限向乙方支付技术服务费用的, 经乙方书面催告后仍不支付的, 每逾期一日, 按照应付费用千分之五的标准向乙方支付违约金, 违约金累计不得超过应付费用的 10%。

3. 乙方无正当理由未按照合同约定期限完成技术服务并向甲方提交服务成果的, 每逾期一日, 按照合同总金额千分之五的标准向甲方支付违约金, 逾期超过三十日的, 甲方有权解除合同, 乙方除退还甲方已支付费用外, 还应按照合同总金额 20% 的标准向甲方支付违约赔偿金, 违约赔偿金不足以弥补给甲方造成损失的, 就不足部分乙方还应赔偿。

4. 乙方提交的技术服务成果未通过甲方验收的, 甲方有权要求乙方在合理期限内重新提交技术服务成果, 乙方重新提交的技术服务成果仍未通过甲方验收的, 视为乙方没有履行能力, 甲方有权解除合同, 乙方除退还甲方已支付费

合同编号：

技术服务合同

项目名称：一种新型超声引导下胸腔穿刺针

委托方：北京大学第三医院

(甲方)

受托方：北京理工大学

(乙方)

签订时间：2022年10月20日

签订地点：北京市

有效期限：2022年10月21日-2022年11月30日

技术服务合同

委托方(甲方): 北京大学第三医院
住 所 地: 北京市海淀区花园北路 49 号
法定/委托人: 乔杰
项目联系人: 杨诗源
联系方式: 13810388916
通讯地址: 北京市海淀区花园北路 49 号

受托方(乙方): 北京理工大学
住 所 地: 北京市海淀区中关村南大街 5 号
法定/委托人: 龙腾
项目联系人: 耿志帅
联系方式: 13621105946
通讯地址: 北京市海淀区中关村南路 5 号
电 话: 13621105946 传真: _____

本合同甲方委托乙方就 一种新型超声引导下胸腔穿刺针制备 项目进行的专项技术服务,并支付相应的技术服务费。双方经过平等协商,在真实、充分地表达各自意愿的基础上,根据《中华人民共和国民法典》(合同编)及相关法律法规要求,达成如下协议,并由双方共同恪守。

第一条: 甲方委托乙方进行技术服务的内容如下:

1. 技术服务的目标: 完成一种新型胸穿穿刺针材料的改进
2. 技术服务的内容: 开发一种新材料或涂层,使穿刺针硬度增加,并具备一定抗菌功能,以便更好地实现穿刺及切割,并预防感染
3. 技术服务的方式: 新材料设计及制备,并初步试制穿刺针

第二条: 乙方应按下列要求完成技术服务工作:

1. 技术服务地点: 北京理工大学材料学院
2. 技术服务期限: 6 个月
3. 技术服务进度: 2022 年 5 月 至 2022 年 9 月,新型材料设计及制

备(已完成)2022年10月至2022年11月,采用新材料初步试制穿刺针

第三条:为保证乙方有效进行技术服务工作,甲方应当向乙方提供下列工作条件和协作事项:

1. 提供技术资料:

(1) /

(2) /

2. 甲方提供上述工作条件和协作事项的时间及方式: /

第四条:甲方向乙方支付技术服务报酬及支付方式为:

1. 技术服务费总额(即合同总金额)为:80,000.00元(大写:捌万元整),其中不包含增值税的净价为77669.90元,增值税税额为2330.10元。

2. 技术服务费明细:包含材料费、设计费、劳务费等

3. 技术服务费支付方式:分期或一次结清

具体支付方式和时间如下:

(1)分期支付:由甲方分期支付乙方,其中首付%

合计元,第二次%合计元,尾款%。

合计元。

(2)一次性支付:检测结束后,甲方收到检测报告并验收合格后7(天)内一次结清。

通过银行转账方式:乙方开户银行名称、地址和帐号为:

开户银行:中国工商银行北京紫竹院支行

地址:北京市海淀区南大街甲10号银海大厦

帐号:0200007609014435495

第五条:双方确定因履行本合同应遵守的保密义务如下:

甲方:

1. 保密内容(包括技术信息和经营信息):本合同涉及的技术服务内容、技术服务要求、技术背景资料、可行性论证报告、技术评价报告、技术标准和规范、原始设计和工艺文件,及各自所有的技术、业务和经营信息等在内的所有信息与资料。

2. 涉密人员范围: 直接或间接涉及本合同的甲方所有相关人员

3. 保密期限: 自合同签订之日起的 1 年

4. 泄密责任: 乙方有权要求甲方停止侵害、消除影响并赔偿由此给乙方造成的一切经济损失,经济损失包括但不限于因甲方泄密给乙方造成的直接损失和乙方为维护合法权益而支出的律师费、公证费、调查费等间接损失。

乙方:

1. 保密内容 (包括技术信息和经营信息): 本合同涉及的技术服务内容、技术服务要求、技术背景资料、可行性论证报告、技术评价报告、技术标准和规范、原始设计和工艺文件,及各自所有的技术、业务和经营信息等在内的所有信息与资料。

2. 涉密人员范围: 直接或间接涉及本合同的乙方所有相关人员

3. 保密期限: 自合同签订之日起的 1 年

4. 泄密责任: 甲方有权要求乙方停止侵害、消除影响并赔偿由此给甲方造成的一切经济损失,经济损失包括但不限于因乙方泄密给甲方造成的直接损失和甲方为维护合法权益而支出的律师费、公证费、调查费等间接损失。

第六条: 违约责任

1. 甲方未按照合同约定向乙方提供技术资料 and 协作事项的,乙方有权要求甲方在合理期限内提供,由此延误的技术服务期限予以顺延。

2. 甲方无正当理由未按照合同约定期限向乙方支付技术服务费用的,经乙方书面催告后仍不支付的,每逾期一日,按照应付费用千分之五的标准向乙方支付违约金,违约金累计不得超过应付费用的 10%。

3. 乙方无正当理由未按照合同约定期限完成技术服务并向甲方提交服务成果的,每逾期一日,按照合同总金额千分之五的标准向甲方支付违约金,逾期超过三十日的,甲方有权解除合同,乙方除退还甲方已支付费用外,还应按照合同总金额 20% 的标准向甲方支付违约赔偿金,违约赔偿金不足以弥补给甲方造成损失的,就不足部分乙方还应赔偿。

4. 乙方提交的技术服务成果未通过甲方验收的,甲方有权要求乙方在合理期限内重新提交技术服务成果,乙方重新提交的技术服务成果仍未通过甲方验收的,视为乙方没有履行能力,甲方有权解除合同,乙方除退还甲方已支付费用外,还应按照合同总金额 20% 的标准向甲方支付违约金。

项目编号



基础产品创新科研项目任务书

所属领域: 火炸药

项目名称:  多维谱指纹鉴定与危险识别

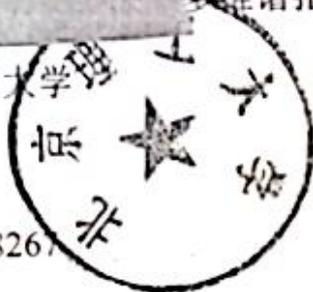
承研单位: 北京理工大学

项目负责人: 李定华

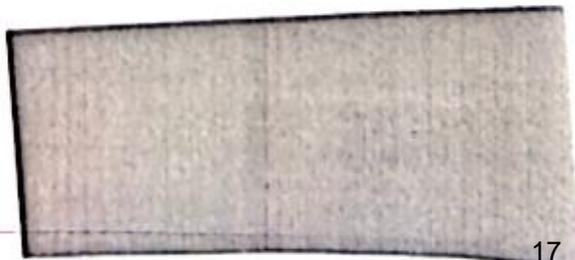
联系电话: 13601038267

研究周期: 2024年1月—2025年12月

编制日期: 2023年12月27日



国家国防科技工业局



赛 Nominate Video Award; 中国轻工业联合会科技进步奖三等奖, 中国电子学会电子信息科技进步一等奖。

表3 主要参研人员情况

姓名	年龄	职称/职务	工作单位	在项目中承担的任务	年工作时间	备注
				检测/分析/识别/预		

耿志帅	32	助理教授	北京理工大学	捏合过程模拟试验	8月	
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研究经费按研究内容进行费用分解，具体见表 11。

表 11 按研究内容的经费预算表

单位：万元

经费类别	金 额				备注
	研究内容一	研究内容二	研究内容三	研究内容四	
材料费	162	0	8	110	
专用费	28.8	11.6	3.6	76	
外协费	50	25	28	161	
燃动费	5	0	0	35	
事务费	13.5	10	11	48.5	
固定资产折旧费	0	0	0	40	
管理费	17.75	7	5	39	
工资及劳务费	45.35	20	12.5	54.4	
不可预见费	0	0	0	0	
收益	10	5	1.5	11.5	
合计	330.4	78.6	71.6	575.4	
说明：表中每个计费项目需对应每一项研究内容的研究方案详细说明测算依据、数量、测算单位、单价以及测算方法等。					

表 11 中相关经费的详细测算说明：

材料费：合计 280 万。拟购置的主要原材料等见表 12。