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**Expanding Polymer Architectures through Metal-Free Ring-Opening
and Ring-Expansion Metathesis Polymerization**

By

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6. Polymer Architecture: Chains, Networks, and Rings

6.1. Introduction

Science is often presented as a collection of established facts, but it is a systematic process for understanding the world around us. I was excited for the opportunity to write this chapter because it gives me a chance to share aspects of my research with family, friends, and other readers who may not have a scientific background. As a polymer and materials scientist, I hope to encourage more people to be curious about the materials and technologies that shape our everyday lives.

The experience of writing for a general audience has also helped me think more carefully about how to organize and communicate complex ideas, a skill that is valuable not only when speaking to the public, but also when communicating with other scientists. Effective science communication also helps people understand how discoveries are made and why they matter. Science communication and science literacy can empower individuals to make informed decisions for themselves and their loved ones, and contribute to a safer, healthier, and more sustainable future.

In this chapter, I will discuss some important properties of polymers and highlight two major research projects from my Ph.D. studies, with the goal of encouraging people to look more closely at the materials that make the modern world possible.

6.2. Part 1: Chains

The Building Blocks of Polymer Chemistry

From water bottles and running shoes to spacecraft and body armor, polymers are everywhere. These materials can be soft and flexible like a rubber band or strong enough to protect against extreme impact. What makes one polymer behave so differently from another? As chemists, we can design polymers with specific properties by choosing the conditions under which they form.

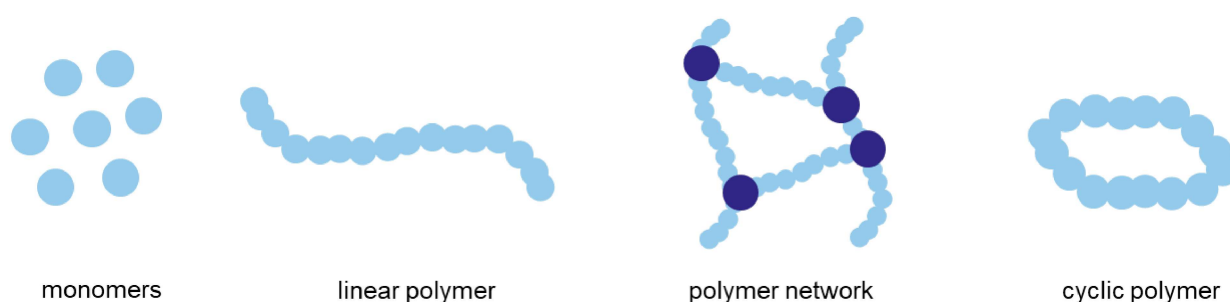


Figure 6.1. Simplified visual representation of monomers, a linear polymer, a polymer network, and a cyclic polymer.

A polymer is a very long chain made of repeating units called monomers. Some chains are short, while others can contain thousands of repeating units. The molecular weight describes the average chain length of a polymer. Like a pile of spaghetti, these chains can become tangled and intertwined. The length of the chains and how they interact determine many of the material's properties. A polymer made of short chains behaves differently from one made of very long chains. Longer chains tend to become more entangled, often leading to stronger and tougher materials. Besides linear chains, polymers can come in many other shapes and configurations, such as polymer networks and cyclic polymers (Figure 6.1).

Scientists can design polymers in several ways: by choosing the type of monomers, controlling how long the polymer chains grow, or arranging those chains into

different architectures. These design choices determine how a material behaves, including how strong it is, how much it can stretch, and how well it performs at different temperatures. Tensile strength describes how much force a material can withstand before breaking, while ductility refers to how far it can stretch before it breaks. The toughness of a material describes how much energy a material can absorb, bend, and stretch without breaking.

Another important property is the glass transition temperature, or T_g . You have probably encountered this phenomenon in everyday life. A plastic container may become brittle in a freezer or feel softer after being heated in the microwave. Although plastic materials are solid, the polymer chains inside are able to move and rotate, depending on the temperature of its surroundings. When the temperature around the polymer is warmer, the polymer chains are more able to move and rotate freely, whereas when it is colder, the polymer chains are less able to move and rotate freely. The temperature range where this shift in behavior occurs is known as the glass transition temperature. By controlling a polymer's structure, scientists can tune these properties to create materials for applications ranging from flexible packaging to protective armor.

In my research, I focused on two architectures in particular: networks, which can be thought of as chains tied together by knots, and rings, where chains form closed loops.

6.3. Part 2: Networks

Connecting Polymer Chains Together in a Crosslinked Network

In polymer chemistry, polymer chains can be connected together in a process called crosslinking. There are two types of crosslinking: physical and chemical crosslinking. Physical crosslinking occurs when the polymer chains form physical knots because they get tangled into each other, like a set of wired headphones in your pocket. Chemical crosslinking occurs when a chemical reaction “glues” the polymer chains together.

Crosslinking is one way to get polymer materials that are both tough and lightweight. The polymer that I worked with during my research is polydicyclopentadiene, or PDCPD. Large durable parts of PDCPD can be molded from liquid components that form a resin formulation. Although the manufacturing process is efficient, the current method of making PDCPD leads to uncontrollable crosslinking. Chemical crosslinks can make a polymer stronger, but too many chemical crosslinks will result in a brittle material. Having fewer chemical crosslinks can lead to a more ductile material that can absorb and distribute energy from bullets or debris more effectively. Additives can be added to modify the ductility of a material, but often adding additives will lead to the material softening at a lower temperature (a lower T_g), which compromises its protective qualities in higher temperature environments.

The first part of my research aimed to improve control over the crosslinking density of PDCPD materials to achieve tougher and higher impact-resistant materials that maintain their mechanical properties at higher temperatures. Our research group, the Boydston group, developed a method to form PDCPD that does not lead to

uncontrollable crosslinking. The method is a light-based polymerization, which uses a photocatalyst and light to start the reaction. This unique method results in PDCPD that allows us to have improved control on the crosslinking that occurs (Figure 6.2). When I added PDCPD synthesized by our method as an additive to a resin formulation to make molded parts, the resulting material is tougher, more ductile, and retains a high T_g .

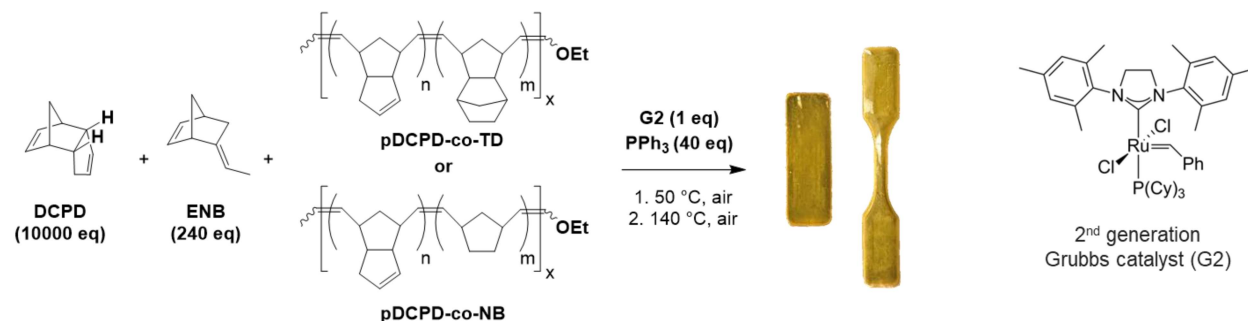


Figure 6.2. Example reaction scheme of making PDCPD samples.

I measured the T_g by using an analytical method called dynamic mechanical analysis, or DMA. With a DMA graph, I identified the T_g by identifying where the storage modulus steadily decreases (the polymer is getting softer) and the temperature where it occurs (Figure 6.3). From these values, I calculated the molecular weight between crosslinks, or M_c . A higher M_c means that there is more space in between each crosslink, indicating a more ductile sample (lower crosslinking density), whereas a lower M_c means there is less space between each crosslink, or a more brittle sample (higher crosslinking density).

I also tracked how the T_g and M_c of the PDCPD molded samples changed over time in a 24-month stability study. The samples were stored in the dark, and at every checkpoint, I performed DMA analysis on the samples. I observed that T_g did not change significantly over 24 months (Figure 6.4). In contrast, M_c decreased significantly over 24 months (Figure 6.5). This suggests that although the PDCPD materials maintain

high T_g , they become more brittle over time (decreasing M_c), most likely due to prolonged oxygen exposure. If the materials contained a surface coating that could prevent oxygen from penetrating, then these materials have potential to be used in more applications.

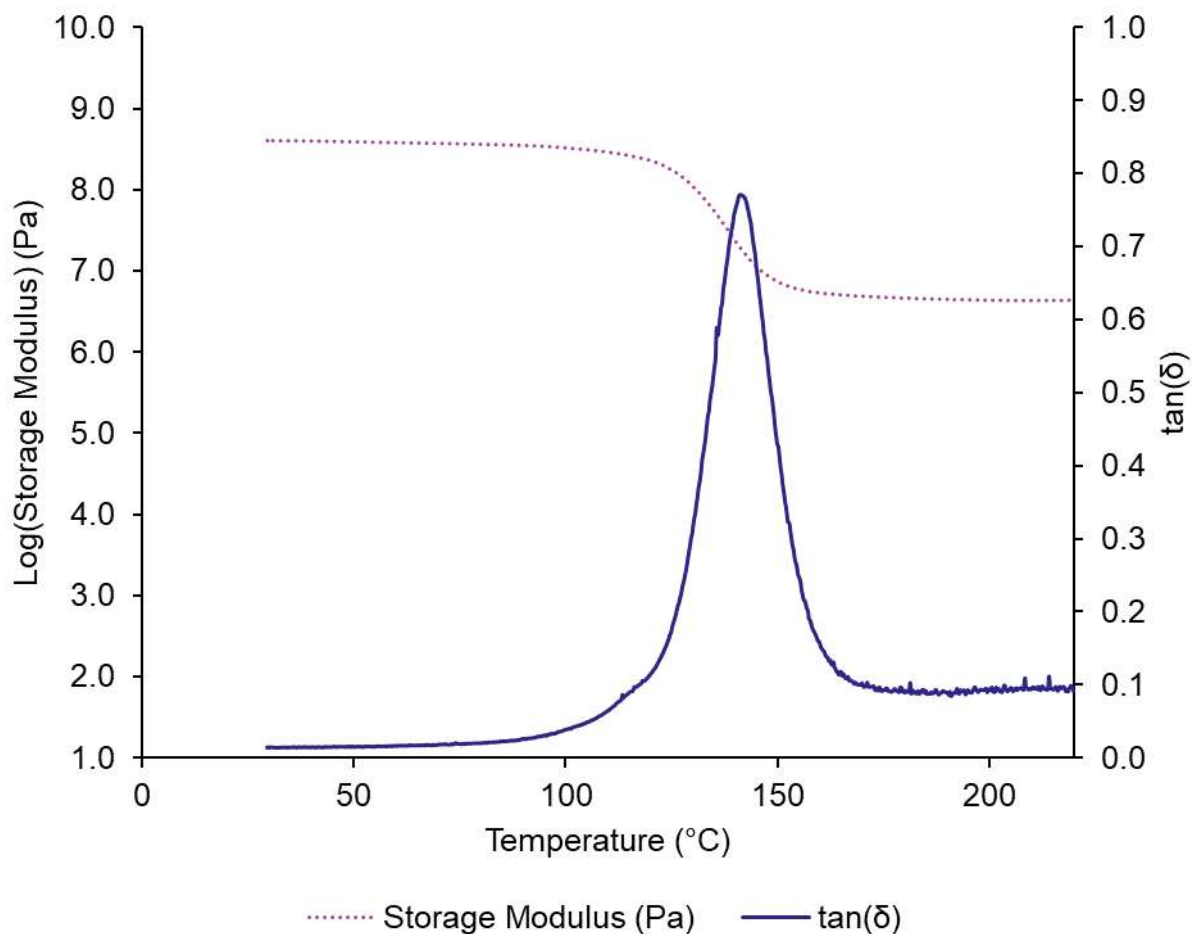


Figure 6.3. Example of a DMA analysis for a PDCPD-based sample.

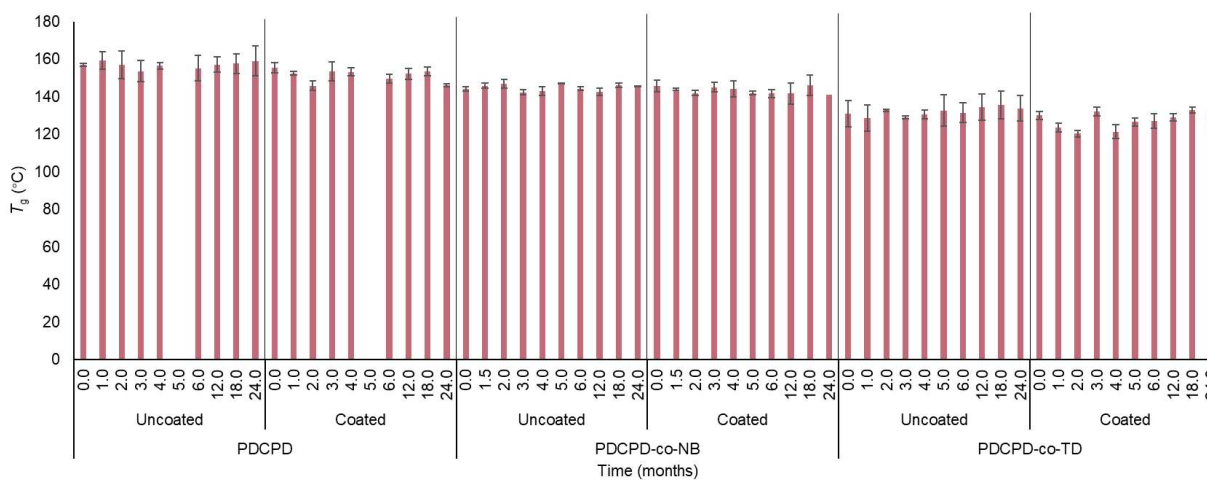


Figure 6.4. T_g over 24 months for PDCPD samples.

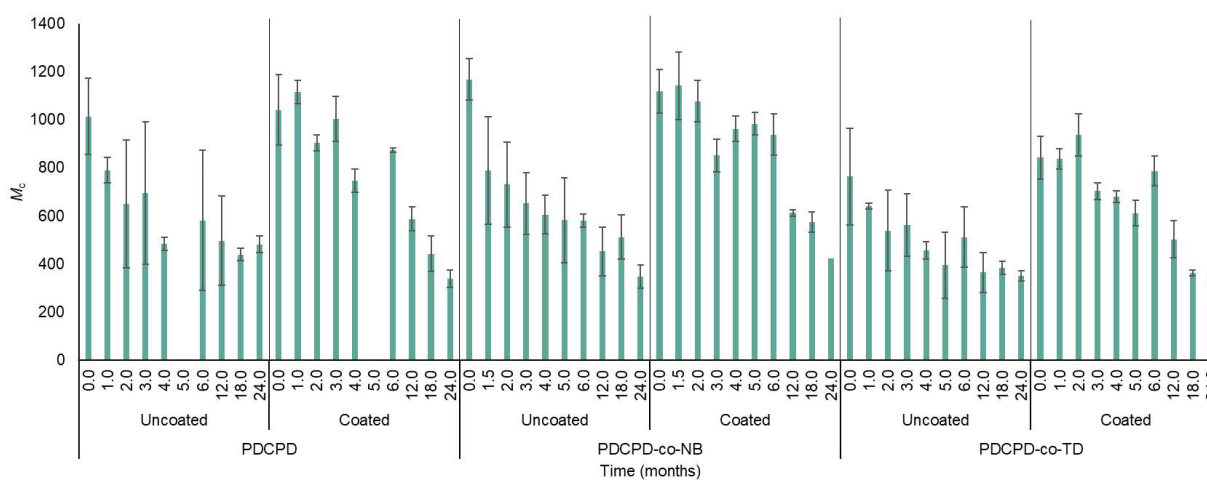


Figure 6.5. M_c over 24 for PDCPD samples.

6.4. Part 3: Rings

Growing Cyclic Polymers

Another way polymer architecture can be modified is by changing the shape of the polymer chains themselves. We can take a linear chain and close the two ends together to make a cyclic polymer. This seemingly small difference in structure can have a surprisingly large effect on how materials behave. For example, cyclic polymers tend to flow more easily upon heating. In technical terms, they have a lower melt viscosity, meaning they can be processed more efficiently when making coatings or molded materials. At the same time, cyclic polymers maintain a high T_g , allowing them to maintain their rigidity at higher temperatures. These properties make cyclic polymers especially promising for applications in coatings, advanced materials, and sustainable manufacturing.

Despite their potential, cyclic polymers are difficult to make. The first approach, called ring closure, starts with a linear polymer chain and connects its two ends together, like fastening a clasp on a necklace. While simple in concept, this process only works efficiently at extremely low concentrations of monomer. Making cyclic polymers by this method requires big reactors and large quantities of solvent to produce small amounts of polymer, which makes it undesirable for industrial-scale production.

A second approach, called ring expansion, takes a different route. Instead of closing a chain, it starts with a small ring and grows it larger by inserting new units into the loop. This can be visualized like expanding a circular train track: a section of track opens, a new piece is inserted, and the loop closes again. Repeating this process creates a larger and larger cycle. This method is generally more efficient, but it often

relies on expensive, complex metal catalysts to start polymerization. These metal catalysts are also difficult to make and have a short shelf life. In both approaches, additional purification steps are often needed to remove impurities to get pure cyclic polymer product.

Our research group wanted to develop a method to produce cyclic polymers more efficiently without needing to synthesize complex metal catalysts. Unlike previous methods that require metal catalysts, our method uses a commercially available, metal-free cyclic enol ether initiator and an organic photocatalyst. This eliminates the need for metals entirely, making our method simpler and potentially easier to scale up than previous methods. The results show that I successfully polymerized 99% norbornene (NB) into cyclic polynorbornene (C-PNB) and nearly 70% DCPD into cyclic pDCPD (Figure 6.6). To our knowledge, this represents the first method for efficiently producing cyclic polymers without the use of metal catalysts.

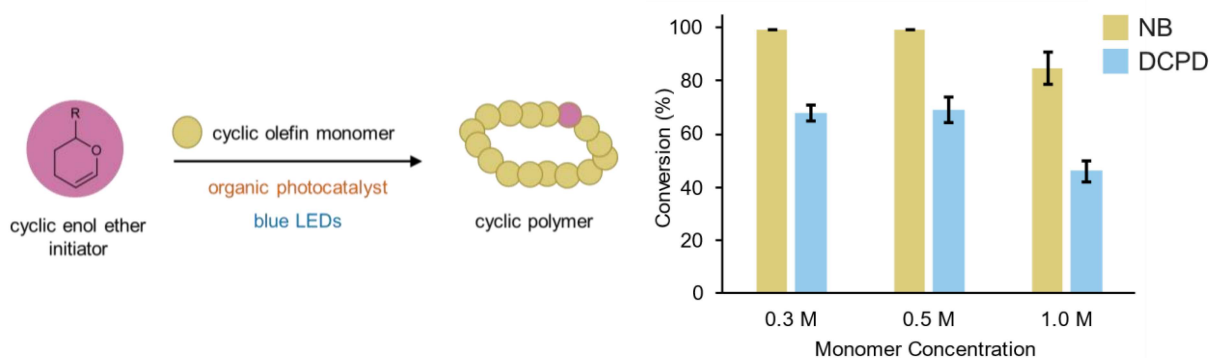


Figure 6.6. Simplified reaction scheme of our method to making cyclic polymers (left). Comparison of the efficiency of our method for making cyclic polymers of norbornene (NB) and DCPD (right).

Looking ahead, cyclic polymers offer a glimpse of a new design strategy in materials science: not just tying or modifying polymer chains, but fundamentally rethinking their shape from the start. As these methods continue to develop, they could enable the next generation of high-performance, sustainable materials.

6.5. Acknowledgements

I would like to thank the Wisconsin Initiative for Science Literacy (WISL) at UW-Madison for providing this opportunity for me to share my research and practice in science communication, and for supporting the development of this chapter. I'd like to specifically thank Professor Bassam Shakhashiri for founding this initiative and Elizabeth Reynolds for the feedback on this chapter.

7. Appendix E: Thin Film Formation of MF-ROMP Products

This appendix contained unpublished work by Trinh, B. K. BKT led the development of spray-coating and water-casting procedures, and performed contact angle measurements. Berthel, E. assisted with the development spray-coating procedures and sample preparation. AJB procured funding and supervised the research.

Overview

We successfully developed a sprayable formulation of polydicyclopentadiene-co-norbornene silane (pDCPD-co-NBSi) in collaboration with BCI. The pDCPD-co-NBSi copolymer contained silane functionality intended to facilitate surface modification and coating performance. We used a carrier solvent system of 80:20 toluene:cyclohexanone for pDCPD-co-NBSi with a commercially available air sprayer to apply multiple coatings of pDCPD-co-NBSi on aluminum and glass substrates. The influence of curing temperature on coating appearance and surface performance was evaluated by contact angle measurements. After undergoing sonication treatment, the contact angles of the samples were measured again, to evaluate coating adhesion and resistance to solvent exposure. A spray-on application will be useful to facilitate product application in the field. Potentially, a sprayable, hydrogenated variant of pDCPD coatings could serve as an oxygen barrier to increase lifetimes of molded parts. Additionally, we describe attempts at water-casting PDCPD solutions to form thin films.