ENGR 240 – Science of Materials Laboratory

LAB T
MECHANICAL PROPERTIES OF CROSSLINKED POLYMER COATINGS

Background

The paints and finishes we use for protection and decoration are polymeric materials—they consist of high molecular weight chain-like molecules, which contain many atoms linked together by covalent bonds. The basic chemical and molecular structure of the polymer (or polymers) from which a coating is made determine many of its important properties, such as hardness, durability, and chemical resistance.

Many common coatings are composed of polymers with a *crosslinked* structure. After application as a liquid, these finishes do not merely 'dry' in the conventional sense. They form hard solid films by undergoing a chemical reaction, called crosslinking (or "curing"), after they have been applied to a surface.

Crosslinking is a process by which linear polymer chain molecules (called "primary chains", because that's what you start with) are joined together at common junction points. These junction points can be provided by smaller (low molecular weight) compounds, which can chemically attach themselves to the ends of at least three different primary chains (see Figure 1). Such compounds are referred to as "crosslinking agents". The structure of a crosslinked polymer can be depicted a net (Figure 1), and these materials are often referred to as *network* polymers.

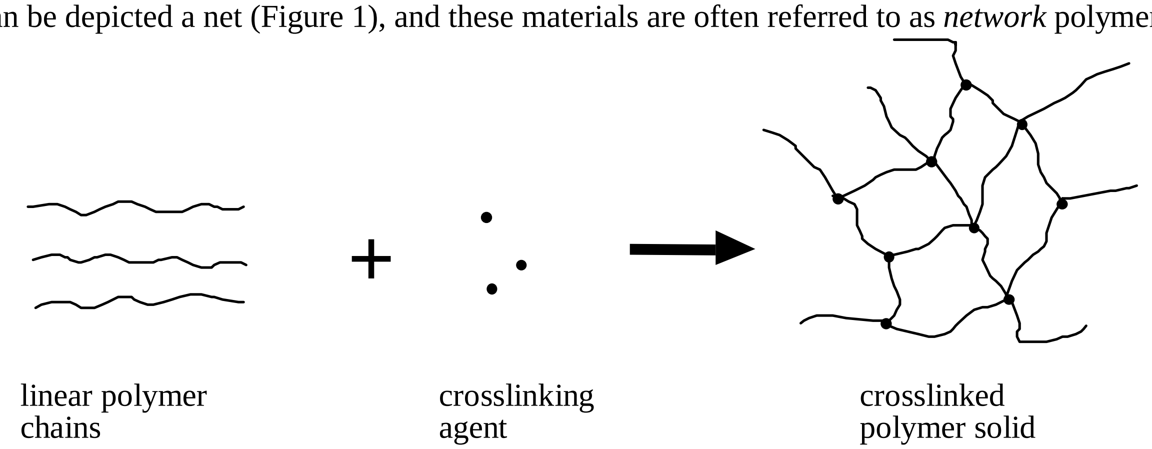


Figure 1. Formation of a Crosslinked Polymer

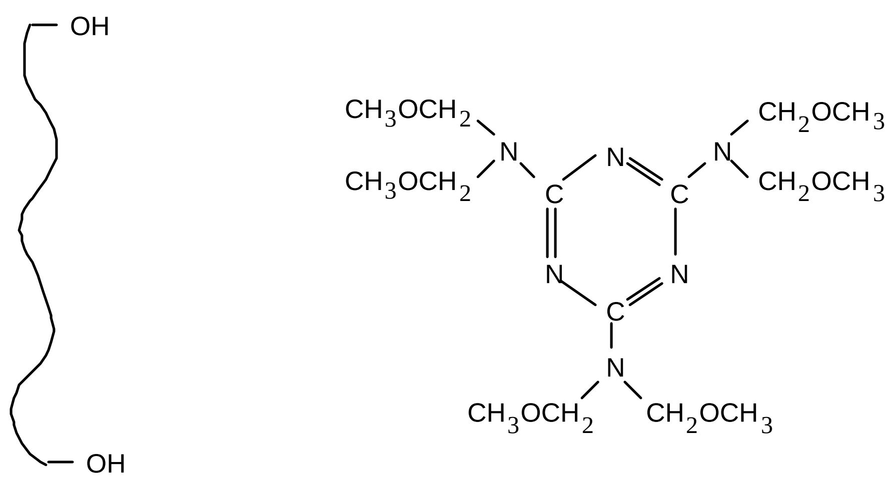
The properties of a network polymer depend strongly on its *degree of crosslinking*. To use the net analogy, a network with extensive crosslinking (many junction points, or many chains emanating from each one) would be classified as a "tight" network, while one with more limited crosslinking would be called a "loose" network. A higher degree of crosslinking in a polymer coating (tighter network) usually leads to increased hardness and solvent resistance, but may also result in increased brittleness. Other mechanical and chemical properties can be affected as well, and usually an optimum degree of crosslinking is sought which gives an attractive combination of properties for a particular application.

<u>In this laboratory, you will prepare a series of crosslinked coatings, and will determine how the degree of crosslinking in these materials affects their hardness and impact resistance.</u>

Some Specifics

Like the schematic of Figure 1, you will fabricate crosslinked polymer networks by combining a primary resin with a crosslinking agent. (The word "resin" is used to describe a polymeric, or high molecular weight, compound). The primary resin consists of linear polymer chains which contain hydroxyl groups (-OH) at the chain ends (see Figure 2). The hydroxyl groups are the reactive sites at which these primary chains will attach to the crosslinking agent. Resins of this type are commonly used for automobile finishes and industrial coatings.

Several different types of compounds can serve as crosslinking agents for the primary chains. The one you will use is a substance called hexamethoxymethyl melamine (HMMM). Its chemical structure is shown in Figure 2.



primary resin chain

crosslinking agent
(HMMM)

Figure 2. Components for Polymer Network
(despite the relative sizes in these drawings, the primary resin chains are in reality much larger than the crosslinking agent molecules, because the chains on the left will be quite long.)

The HMMM chemical structure may appear complex, but the important thing to note is that it contains <u>six</u> (CH_3OCH_2-) groups. These are the chemical groups that can react with the (-OH) groups of the primary resin chains. Such a reaction will result in a connection between the HMMM and the end of a primary chain (this is kind of like putting together Tinkertoys®!). The HMMM can attach itself to as many as six different primary chains, and can therefore serve as junction points for the network (see Figure 1).

A small amount of catalyst and elevated temperatures are needed for the reaction to take place. After the components (primary resin, crosslinking agent, catalyst and some solvent) are applied to a surface as a liquid film, it will be heated in an oven for a specified time. You may have heard of automobile finishes being "baked" in this fashion.

The degree of crosslinking, and therefore the final properties, of the cured films can be controlled by the crosslinking time and temperature (increasing time or temperature leads to higher degrees of crosslinking).

Two important coating properties you will measure for your films, using standardized coating industry tests, are hardness, and impact resistance (a measure of how brittle the films are). For hardness, you will attempt to scratch the films with pencil leads of differing hardnesses.

This type of measurement is particularly appropriate for these materials, because coatings in service may have to resist scratching or gouging by hard objects.

For the impact test, you will drop a weight onto coated steel panels. This test is fairly simple, and again simulates potential service conditions for these materials.

Procedures

1. Mix components:
   - Start with approximately 8 g of the Polyester Resin solution. This solution contains the primary chains (which happen to consist of a type of polymer called a "polyester"), dissolved in a volatile solvent. The concentration of Polyester Resin in this solution is 50 wt%.
   - Add HMMM crosslinking agent to achieve a 1.6:1 target ratio of Polyester Resin (not counting the solvent!) to HMMM.
   - To this final mixture, add 1% by weight of the catalyst (K-Cure 1040).
   - Mix thoroughly.

2. Prepare films:
   - Use the drawdown bar to prepare uniform, constant thickness films on steel panels. Check with instructor for recommended film thickness and number of samples.
   - Allow panels to remain in the hood for 10 minutes to allow for solvent evaporation.

3. Crosslink films:
   - Place samples in ovens at specified temperatures for 30 minutes. Check ovens periodically for temperature maintenance.

4. Analyze films:
   - Touch films to see whether sufficient reaction occurred (compare to an unheated film).
   - Using the microhardness tester, determine the film hardness at each crosslinking temperature.
   - Using the drop weight tester, investigate the films' resistance to impact at each crosslinking temperature. Report the "impact energy" (in inch-pounds) as the highest energy at which the sample consistently <u>does not</u> fail.

Report:

Include the following in your report:

- Plots of film hardness and impact energy as a function of crosslinking temperature.
- Discussion of the effects of temperature on mechanical properties, including expected results for mechanical properties that were not tested (such as ductility and strength).

References:

- Callister Chapters 15 and 16.