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#23 Glass Transition in a Rubber Ball

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I. INTRODUCTION

Description

This experiment provides a dramatic way to illustrate the changes in the properties of a material at its glass transition point. Students gather data which they use to construct graphs to learn about elastic modules versus absorption modulus, tangent delta, and the effect of impact speed on the glass transition temperature.

Student Audience

This activity could be done in high school chemistry classes through university level chemistry or in a chemical technology course.

Goals for the Experiment

While doing this experiment, the student will:

- measure rebound height as a function of temperature,
- calculate the difference between drop height and rebound height as a function of temperature,
- plot both rebound height and the difference between drop height and rebound height as a function of temperature,
- relate rebound height to elastic modulus and the difference between drop height and rebound height to absorption modulus,
- calculate tangent delta and relate it to glass transition temperature,
- compare glass transition temperature for two balls of different rubber composition and discuss implications for use, and
- predict the effect of impact speed on glass transition temperature and discuss implications for tire manufacturing.

Recommended Placement in the Curriculum

This experiment is appropriate during a discussion of any of the following topics:

- kinetic and potential energy,
- energy transformation,
- conservation of energy,
- phase changes, and
- physical properties of materials.

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II. STUDENT HANDOUT

Glass Transition in a Rubber Ball

Scenario

Your company, Stretch Corporation, sells various formulations of rubber to tire and other manufacturers. The Research Department has developed several new formulations which need to be tested prior to large-scale production. Their properties need to studied to see if there might be a market for either or both. As a company technician, you are asked to determine the glass transition temperatures of the samples.

Background

In studying the glass transition temperature, polymer scientists consider the absorption (or loss) modulus (a measure of the absorption of energy) and the elastic modulus (a measure of the ability to return to an initial form after deformation) of a polymer. The rebound height of the rubber ball is proportional to the elastic modulus and the difference between drop height and rebound height is proportional to the absorption modulus. The ratio of these terms is “tangent delta” and is an indicator of second order transitions such as glass transition. At the glass transition temperature, tangent delta displays a peak. This means that below the glass transition temperature the material is chiefly elastic in the sense that a glass marble bounces. Above the glass transition temperature the material would be a liquid if it were not chemically crosslinked to form rubber. At the glass transition temperature, the material is almost completely absorbing leading to properties similar to lead.

Because the glass transition temperature is not a true thermodynamic transition it has kinetic features meaning that it changes with the speed of the ball at impact. This can be accomplished by increasing the height from which the ball is dropped. Such shifts with the impact speed can be accounted for theoretically. Tangent delta is used in the tire industry as a measure of adhesion performance of a tire. Generally a broad tangent delta is desired for good adhesion at variable driving speeds.

Safety, Handling, and Disposal

- Wear safety goggles during the experiment. There is a possibility that the frozen rubber balls may shatter when dropped.
- All laboratory work should be done in a well ventilated room.
- Be sure to have no open flames in the vicinity, as they may ignite the acetone vapor.
- Use tongs to handle pieces of dry ice. Avoid direct contact with the skin.
- Acetone used in baths can be reused. Return to instructor when finished.
- Remove all acetone from work area before using hot plates.

Materials

- safety goggles
- meter stick or metric tape
- super ball and stupid ball (a.k.a. happy ball and sad ball)
- masking tape
- dry ice
- ice

- acetone
- 400-mL beaker
- 500-mL acetone wash bottle filled with acetone
- crucible tongs
- -100–50 °C thermometer
- -20–110 °C thermometer
- hot plate

Procedure:

Investigation #1: The Super Ball

1. Tape the meter stick or metric tape to a wall or flat vertical surface so that the zero mark touches the floor or table top. (The floor or table top must be a rigid material in order to serve as the rebound surface.)
2. Drop the super ball from a height of 1 meter and record the height of the rebound.
3. Wearing goggles, place the super ball in the 400-mL beaker and ask your instructor to add the dry ice/acetone mixture until it covers the ball. Check the temperature in the bath with the -100–50 °C thermometer. If the temperature does not read about -78 °C, use the tongs provided to slowly add small pieces of dry ice while gently stirring with the thermometer. If necessary, keep adding more dry ice in order to maintain this temperature. Allow the super ball to remain in the dry ice/acetone bath for 5 minutes. Record the bath temperature.
4. Remove the super ball from bath and immediately drop from a height of 1 meter and record the rebound height. Return the super ball to the bath.
5. Raise the bath temperature by about 10 °C by adding a small amount of room temperature acetone from the 500-mL wash bottle. Record this temperature. Keep the bath at this temperature for 5 minutes before removing the super ball from the bath and immediately dropping it from a height of 1 meter. Record the rebound height and return the super ball to the bath.
6. Repeat Step 5 until the bath temperature is between -10 °C and +10 °C. After dropping the super ball and measuring the rebound height, return the acetone bath to your instructor and replace it with an ice water bath. You will also need to replace the thermometer with the one that has a -20–110 °C range.
7. Raise the temperature of the water bath by adding warm water until it reaches about 10 °C. Wait 5 minutes and then remove the super ball from the bath, immediately drop it and record the rebound height. Continue to do so in 10 °C increments until the rebound height is close to that measured in Step 2 or the bath temperature is at room temperature.
8. For each temperature, the rebound height is proportional to the elastic modulus. Calculate the difference between the drop height (1 meter) and the rebound height. This is proportional to the absorption modulus. Finally, calculate the “tangent delta,” the ratio of this difference to rebound height.
9. Plot rebound height versus temperature and on the same graph the difference between drop height and rebound height versus temperature. Construct a second graph in which tangent delta is plotted versus temperature. Tangent delta displays a peak at the glass transition temperature. Record this temperature.

Investigation #2 The Stupid Ball

1. Replace the super ball with a stupid ball.
2. Drop from a height of 1 meter. Place the stupid ball in a dry/ice acetone bath around -40 °C and allow it to remain in the bath for about 5 minutes as before
3. Remove the super ball from the bath and drop from a height of 1 meter. Record the rebound height. Raise the temperature of the bath as in Investigation #1, dropping the ball and measuring the rebound height. Repeat until the temperature reaches 0 °C and then switch to an ice water bath. Repeat testing at 10 °C intervals but this time do not stop heating at room temperature. Instead, place the water bath on a hot plate, heat, and continue testing the ball at 10 °C intervals until a temperature of 80 °C is reached. Be sure to remove all acetone from your work area before turning on the hot plate.

2. As with the super ball, calculate the difference between the drop height (1 meter) and the rebound height. Also calculate the tangent delta, the ratio of this difference to rebound height.

3. Construct a new graph for the stupid ball by plotting rebound height versus temperature and the difference between drop height and rebound height versus temperature. However, plot tangent delta versus temperature for the stupid ball on the same graph as the super ball. Determine the glass transition temperature for the stupid ball and record it.

Questions

1. Describe how the balls bounce a) below the glass transition temperature, b) at the glass transition temperature, and c) above the glass transition temperature.
2. For each rubber ball, give the temperature where it shows the best adhesion (least rebound).
3. Since glass transition temperature is not a true thermodynamic property, predict the effect of increasing the impact speed on the glass transition temperature. If time permits, repeat the experiment with either or both balls but increasing the drop height to 2 meters.
4. If you were buying a tire, what features would you look for in the choice of rubber used?
5. If you had a bunch of used golf balls and tennis balls, how would you pick out the best ones?

References

1986 Annual Book of American Society for Testing and Materials Standards, Volume 9.01 "D-2632-79 Standard Test Method for Rubber Property—Resilience by Vertical Rebound"; ASTM: Philadelphia PA, 1986; pp. 612-613.

Shah, V. *Handbook of Plastics Testing Technology,* John Wiley & Sons: New York, 1984.

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III. INSTRUCTOR NOTES

Glass Transition in a Rubber Ball

Purpose

In these investigations, you will observe the effect of temperature changes on the ability of several rubber balls to bounce. The data you gather will allow you to calculate tangent delta and thus determine the glass transition temperature for the rubber in the ball. You will compare glass transition temperatures for balls having different rubber compositions and may observe how this temperature changes as a result of impact speed.

Time Required

This activity can be performed in 1-2 hours.

Suggested Group Size

This activity is best completed with students working in pairs. Any size group can do this as long as there is wall and floor space available.

Materials

Per pair of students

- safety goggles
- meter stick or metric tape
- super ball and stupid ball (Happy ball and Sad ball; One supplier for 1.5-inch balls is Hawkeye Rubber of Cedar Rapids, IA; (319) 363-2679. The balls are also sold as Smart/Stupid Balls (AP 1971) by Flinn Scientific Inc., P.O. Box 219, Batavia, IL 60510-0219; (800) 452-1261. Whamo Mfg. of San Gabriel, CA. makes 1-inch balls.)
- 400-mL beaker
- 500-mL acetone wash bottle filled with acetone
- crucible tongs
- -100–50 °C thermometer
- -20–110 °C thermometer
- hot plate

Per class

- 2–3 liters of low grade acetone
- 2–3 liter insulated container or dewar
- 2–3 lbs. of dry ice
- 1 bag ice
- 1 roll masking tape

Safety, Handling, and Disposal

- Wear safety goggles during the experiment. There is a possibility that the frozen rubber balls may shatter when dropped
- All laboratory work should be done in a well ventilated room.
- Be sure to have no open flames in the vicinity, as they may ignite the acetone vapor.
- Caution students not to touch dry ice. Use forceps to pick up pieces of dry ice. Avoid direct contact with the skin.

- Remind students to return acetone to instructor when finished with bath for each activity. Acetone used in baths can be reused. This will also minimize the possibility of a hot plate causing the acetone to ignite. Use care here. The acetone is highly carbonated by this procedure and pressure will build up if the acetone container is stoppered.

Points to Cover in Pre-Lab

- This lab should follow the investigation "Simplified Vertical Rebound Testing" by Melanie Stewart in which students perform vertical rebound tests on different types of rubber balls. This way they will already be familiar with the concept of energy transfer, have measured rebound heights, and have calculated the difference between drop height and rebound height.
- If your students have already done the rebound tests on different types of balls they may already wonder about the difference in composition of rubber balls and which would be most suitable for use in other products such as tires. This lab will help them to "discover" the behavior of rubber at its glass transition temperature and tangent delta and its importance to the adhesion performance of tires at variable driving speeds.
- While they perform the experiment, encourage students to make note of key assumptions and sources of error. These need to be discussed in the post lab discussion.

Procedural Tips and Suggestions

- You may want to use question 5 as a starting place for the investigation.
- You may prefer to have the students start their measurement at room temperature and then go down (and later up) from there. They may find it easier and quicker to start high and gradually add dry ice to the acetone bath to lower the temperature. This approach uses more dry ice but less acetone.
- If possible, the night before the experiment is to be done, place the bottle containing the 2–3 liters of acetone for the bath in an explosion proof freezer. This will reduce the amount of dry ice needed initially to lower the bath temperature to a minimum. Editor's note: One of the reviewers suggested that this not be done. She indicated that it is not necessary to use much dry ice to lower the temperature of the acetone.
- Right before the students are to perform the experiment, place the cooled acetone in a wide mouth dewar or insulated container and add dry ice until the temperature is reduced to the lowest temperature at which dry ice sublimes, approximately -78 °C.
- Have students bring their 400-mL beaker to the front desk when ready and pour out just a little more acetone than is needed to cover the ball.
- Initially students may need to add more dry ice to their bath. Make sure they add small pieces slowly, gently stirring as they add them to the bath to prevent too much gas being released from the dry ice at one time.
- Remind students that they need to leave the ball in the bath for 5 minutes at the desired temperature to allow the ball to reach that temperature. When removed from the bath, bounce it as quickly as possible and then return it back to the bath.
- Not all super/stupid balls will work in this experiment. Some tested would not reach their glass transition temperature by cooling in a dry ice/acetone bath. Those made of black, neoprene rubber work best because of their high density (sink in water) and relatively high glass transition temperatures. Those low density super balls made of butyl rubber have glass transition temperatures well below that obtainable in a dry ice/acetone bath. Either the large neoprene balls (1.5-inch in diameter) or the smaller (1-inch diameter) balls can be used. The smaller, 1-inch diameter balls will tend to heat up much faster.
- It may be possible to reduce the 5 minute cooling period to, for example, 3 minutes. You should,

however, try the shorter time with the particular balls your students will use in the investigation.

- You can cut down the time for this investigation as well as the amounts of dry ice and acetone needed by having groups share data. The easiest way to do this is to have some groups investigate the super ball and others the stupid ball. You could also divide up the temperatures but this has the potential for introducing error due to slightly different techniques used in, for example, reading the rebound height.

Sample Results

Temperature (° C)	Rebound Height (cm)	Difference (cm)	Tangent Delta
–78	28	72	2.5
–68	22	78	3.5
–59	20	80	4.0
–36	7	93	13
–24	4	96	24
–14	6	94	16
–5	10	90	9.0
3	18	82	4.6
13	29	71	2.4
24	48	52	1.1

Plausible Answers to Questions

1. Describe how the balls bounce a) below the glass transition temperature, b) at the glass transition temperature, and c) above the glass transition temperature.

A. When compared at room temperature, the super ball and stupid ball seem very dissimilar—the former is a great bouncer and the latter has little if any bounce. However, when compared relative to their individual glass transition temperatures, they show remarkably similar bouncing characteristics. The amount of rebound is minimal at the glass transition temperature (near room temperature for the stupid ball) and increases at temperatures both above and below

2. For each rubber ball, give the temperature where it shows the best adhesion (least rebound).

A: This occurs where tangent delta is a maximum, i.e. the glass transition temperature. Some students may list only a single temperature for each ball while others will give a range. The temperature (range) should correspond to their data.

3. Since glass transition temperature is not a true thermodynamic property, predict the effect of increasing the impact speed on the glass transition temperature. If time permits, repeat the experiment with either or both balls but increasing the drop height to 2 meters.

A: It's hard to say how they will answer this question. In theory, as the impact speed increases, more energy will be absorbed, shifting the glass transition temperature to a higher value. On the other hand, if they base their answer on a experiment which suggested increasing the drop height to 2 meters (impact speed will increase 1.4 times) they may not have the data to support this conclusion and instead say it makes no difference. A lot depends on the skill of the experimenter.

4. If you were buying a tire, what features would you look for in the choice of rubber used?

A: Answers may vary but hopefully included will be a statement about a broad tangent delta at variable temperatures and driving speeds.

5. If you had a bunch of used golf balls and tennis balls, how would you pick out the best ones?

A: Transfer of kinetic energy (as measured by rebound) to each of these balls is very important. A quick test which players use is just to bounce the ball and see how well it rebounds. The greater the rebound, the better the ball. A more quantitative test would be to determine the relative rebound heights.

Extensions and Variations

See suggestion in Question #3 to repeat the experiment with either or both balls but instead increasing the drop height to 2 meters.

References

1986 Annual Book of American Society for Testing and Materials Standards, Volume 9.01 "D-2632-79 Standard Test Method for Rubber Property—Resilience by Vertical Rebound"; ASTM: Philadelphia PA, 1986; pp. 612-613.

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