

Phase change experiment: Nitinol and Bobby Pins

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Abstract:

In this experiment, students are introduced to the effects of changes in crystal structure (“phase”) in metals, specifically in high carbon steels and in the shape memory alloy “Nitinol.” Phase changes in both of these materials cause changes in properties (for steels) and shape (for Nitinol).

First the students see the effects of a phase change in Nitinol through a demonstration. Then the students see the effects of steel heat treatment by heating and cooling bobby pins to form the hard and brittle phase, Martensite. These effects are specifically related to processing parameters, and these examples show the influence of properties on processing, as influenced by structure.

Objectives: Students will be able to

Explain the effect of phase changes on shape in Nitinol

Experience the effects of crystal structure changes on metal properties

Show the effect of phase changes on strength in steels

MatEd core competencies addressed (most important in bold):

0B Prepare tests and analyze data

4A Demonstrate effective work with teams

8A Demonstrate the planning and execution of materials experiments

9A Classify constituents, properties and processing of steel

9G Discuss advantages of Nickel alloys and their uses

9H Compare uses and processing of Titanium and its alloys

Key words: shape memory, phase change, Nitinol, steel, heat treatment

Type of Module: Demonstration plus classroom experiment

Time required:

Nitinol demonstration: 5 - 10 minutes

Bobby Pin Experiment—

Introductory classes: 45 minutes with discussion

Advanced classes: 30 minutes with discussion

Grade Levels: Advanced high school and college undergraduate

Equipment and supplies needed

Part 1:

Nitinol wire

Means of boiling water (using a beaker or coffee cup)

Part 2:

Supply of standard bobby pins (the springy kind)

Means of heating the bobby pins (bunsen burner or propane torch or similar)

Tongs

Beaker of water

Instructor background and notes:

In certain specialty alloys, phase changes can cause the metal to remember its former shape. This is seen specifically “shape-memory alloys” such as the nickel-titanium alloy called “Nitinol.” Nitinol (an acronym for Nickel Titanium Naval Ordnance Laboratories where the alloy was discovered) is an alloy of nickel and titanium in different combinations; Nitinol to be used in this experiment should be 50-50 Ni-Ti, which gives it a transformation temperature below 200F (93C).

Part 1 of this experiment is based on a demonstration of Nitinol. In Nitinol, the phase change that causes the shape-memory effect is a face centered cubic (FCC) to monoclinic phase change, very similar to the Austenite (FCC) to Martensite (monoclinic) phase change in steels. Please see reference 1 for sketches of the actual crystal structures, which are similar except for the types of atoms. For Nitinol, the Ti and Ni atoms are close to the same size. Both high temperature and low temperature structures are ductile and easily bent.

Part 2 of this experiment uses steel. Steels are alloys of iron with up to 1% carbon and exist in two important crystal structures: Body Centered Cubic (BCC) at room temperature and up to 1333F (723C), then the structure changes to Face Centered Cubic (FCC). This change in structure, also called a phase change, is the key to heat-treating of steels because BCC dissolves only a small amount of carbon, with most of the carbon precipitating as a second phase, iron carbide. This is the case in standard grade construction steel, which consists typically of iron plus 1 weight percent carbon, where the C atom is much smaller than the Fe atom.

The FCC phase has a much larger solubility limit for carbon so the carbon exists in a solid solution at the high temperature. When high carbon steel is cooled from the FCC phase, two things can happen:

- Slow cooling will cause the precipitation of the iron carbide phase, and the material will be strong but not brittle; or
- Rapid cooling will prevent the precipitation of the carbide phase because not enough time is available for the atoms to arrange themselves in the carbide phase.

In the rapid cooling case, there are too many carbon atoms so they cause the formation of Martensite, a distorted monoclinic structure. In steels, this phase is very hard and brittle and will break before it bends.

IMPORTANT: It should be noted that heat treatment is fundamentally different from the process called “annealing.” Heat treatment effects come from a change in crystal structure of the steel. Annealing does not involve a crystal structure change. Rather, annealing at a high temperature, which makes a cold worked metal soft, comes from removing defects from the metal and causing the formation of a new, strain-free structure. A variety of experiments are available that demonstrate the effects of annealing.

In summary, we use Nitinol as the model for the phase change in steel in this experiment. Since the crystal structures are similar and are referred to by the same names, Austenite and Martensite, this provides a uniform base for student understanding. However, the properties are quite different because the alloys are different:

- For Nitinol, the Ni-Ti alloy, the alloy is formed at high temperature, where it exists as Austenite, and remembers the shape in which it was formed. When cooled, it transforms to the Martensite phase. Here, the Martensite phase is quite flexible since the Ni and Ti atoms are of a similar size, and this Martensite structure can be bent into any desired shape. Re-heating into the FCC range, however, the wire remembers its shape when it was Austenite and reverts to that shape. This is a good, gee-whiz demonstration to get the students interested and to teach the differences in phases of metals. There are also other materials that demonstrate this property, but Nitinol is the most available.
- For steels, which are alloys of iron and carbon, the phases are the same but the properties much different. Steel at room temperature exists in a BCC structure called Ferrite. When heated, it transforms to FCC Austenite. When cooled very rapidly, it transforms into Martensite, which is a very hard and brittle phase. Referring again to the Martensite crystal structure as above, the additional atoms in the structure are very small carbon atoms, which put the structure under stress, hence the brittle behavior as demonstrated in part 2 of this experiment.

For a more advanced module on steels, please refer to the MatEd module, "Iron and Steel—Properties and Applications" listed in the references, below.

Part 1, Nitinol

Here we start at room temperature (Martensite), where the Nitinol wire can be bent into a shape as desired. Bending around a finger is the easiest to start with. Then on heating to 200F, the wire will revert to its original shape; on cooling the experiment can be repeated. Note that bending too much causes permanent deformation, and that sharp bends should be avoided.

Procedure

If sufficient Nitinol is available, groups of students can experiment with the wire after the initial demonstration.

1. Obtain a 6 - 8 inch length of Nitinol wire.
2. Heat some water to the range 150 to 200F—keep it close to boiling temperature for the fastest transformation.
3. Gently bend the wire around your finger to make a spring-like structure; show the shape that you have made to the students (such as on an overhead projector)
4. Heat the wire in the hot water—observe the change in shape on heating; show the new shape of the wire
5. Discuss possible applications of this effect

Note: In fact, this effect is used in orthodonture, stents to open up arteries, some eyeglasses and in a wide variety of sensors and toys. In fact, there is a good possibility that many of the students have used items based on Nitinol (and perhaps not known what it was).

Part 2: Bobby Pins

Bobby pins are made of a high carbon steel to provide strength and slow cooled to provide enough ductility so that the pins will not break on use. In this experiment, we test the as-received properties of a bobby pin, and then we provide several types of heat treatment. The properties will vary after each treatment, especially with the heat and quench treatment that will produce Martensite.

Procedure:

1. Each group of students should have 5 bobby pins.
2. Hold one bobby pin by the ends and pull to open it up into a straight line. Bend it back. This is one bend. Count and record the number of bends needed to break the bobby pin.
3. Heat the second bobby pin at its sharp bend until it is red hot. Let this bobby pin cool slowly in air.
4. When the bobby pin is cool, bend it back and forth as before. Count and record the number of bends needed to break this heated and slow cooled wire.
5. Fill the beaker with cold water.
6. Heat the third and fourth bobby pins in the flame until they are red hot and immediately plunge it into the water in the beaker.
7. When these bobby pins are cool, bend number 3 as before and record the number of bends needed to break it. Save the other heat treated bobby pin for process 8, next.
8. Heat the fourth bobby pin again but cool it slowly in air. This process is called tempering and occurs after Martensite is formed. As before, bend the tempered wire and record the number of bends needed to break it.
9. You now have 5 bobby pins, one original and four broken. Compare the results. Discuss the differences and the effects of heat treating and tempering on the properties of this steel.
10. Discuss also what differences you might have seen if:
 - Bobby pin #2 had been quenched instead of slow cooled;
 - Bobby pin # 3 had not been heated enough to make it into the FCC phase;
 - Bobby pin #4 had been reheated until red hot then quenched in water

11. These are effects that might be seen if improper processing procedures had been followed. How important could these errors have been on the performance of a similar steel?

Further study:

Using the Internet, find and explain at least 2 applications of Nitinol used in dentistry, medicine and/or toys

Using the Internet find the difference between heat treating and annealing a metal. Explain the differences. Can you find an annealing experiment that would show the differences?

References

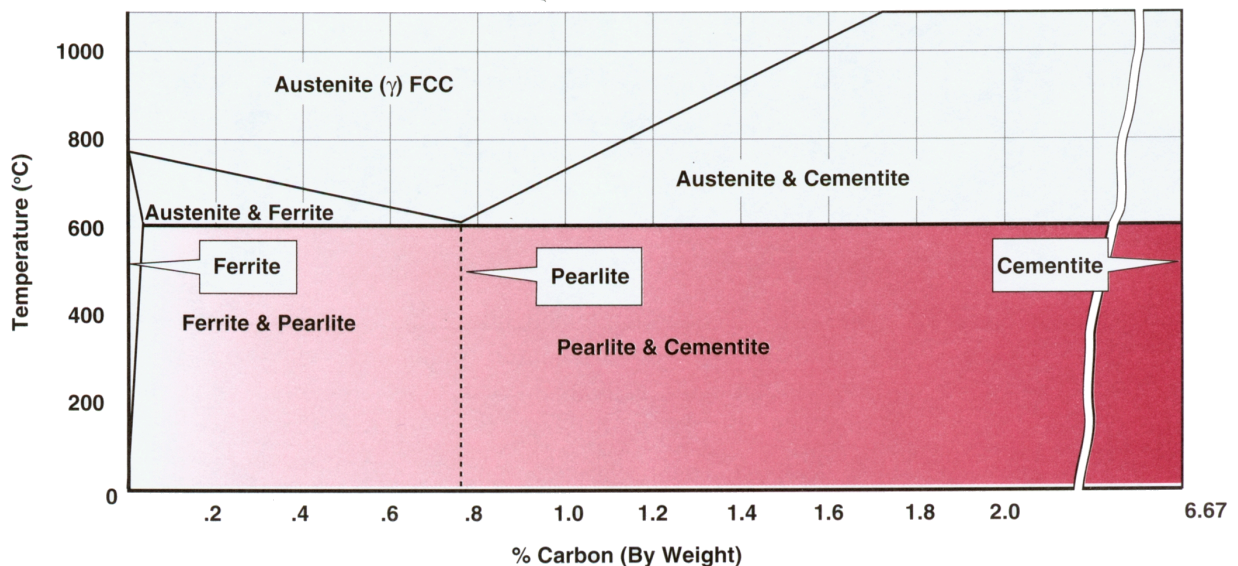
Nitinol:

1. For a sketch of the two Nitinol crystal structures, see http://upload.wikimedia.org/wikipedia/en/e/eb/Nitinol_Austenite_and_marten_site.jpg
2. For a technical explanation, http://en.wikipedia.org/wiki/Shape_memory_alloy
3. For history, <http://www.stanford.edu/~richlin1/sma/sma.html>
4. For a source, <http://www.teachersource.com/catalog/index.html>

Steel:

5. Heat Treatment: http://en.wikipedia.org/wiki/Heat_treatment
6. Iron and Steel—Properties and Applications, <http://www.materialseducation.org/educators/mated-modules/>

Handout for advanced classes: Iron Carbon Phases



A portion of a simplified iron-carbon binary phase diagram

Reference: Energy Concepts, Inc., Len Booth, 1997, ISBN #1-55756-167-2

Various Alloys and Phases of Iron and Steel

Name	% Carbon (by weight)	Characteristics
Ferrite	0-0.025	BCC iron; fairly soft and ductile
Pearlite	0.8	A mixture of ferrite and cementite
Cementite	6.67	Iron carbide, Fe ₃ C; Very hard and brittle
Austenite	0-2.11	FCC form of iron and steel; Normally occurs only at temperatures >727°C
Martensite	0.02-2	Made by rapidly quenching austenite; Hard and brittle
Cast Iron	2-5	Very brittle; Not considered steel

Evaluation:

Student evaluation questions (discussion or quiz):

1. What caused the Nitinol to go back to its original shape?
2. Why does the bobby pin not go back to its original shape?
3. What causes the fast cooled bobby pin to break? Why?
4. "The fact that steel can be heat treated makes it a very important material in our society." Explain this statement and give examples.

Instructor evaluation questions:

1. At what grade level was this module used?
2. Was the level and rigor of the module what you expected? If not, how can it be improved?
3. Did the activity work as presented? Did they add to student learning? Please note any problems or suggestions.
4. Was the background material sufficient for your background? Sufficient for your discussion with the students? Comments?
5. Did the activity generate interest among the students? Explain.
6. Please provide your input on how this module can be improved, including comments or suggestions concerning the approach, focus and effectiveness of this activity in your context.

Course evaluation questions (for the students)

1. Was the activity clear and understandable?
2. Was the instructor's explanation comprehensive and thorough?
3. Was the instructor interested in your questions?
4. Was the instructor able to answer your questions?
5. Was the importance of materials testing made clear?
6. What was the most interesting thing that you learned?