ME 280 Lab #1

Lüders Strain Evaluation of Stress Concentration
and
Electrochemical Embrittlement of Spring Steel

Overview:

In one portion of this lab, you will observe the propagation of a Lüder’s band in a tensile bar of annealed low carbon steel, noting the stress needed to propagate the Lüder’s band. Subsequently, you will test a coupon specimen with a central ¼” diameter hole that induces a theoretical stress concentration of three at the edge of the hole. The Lüder’s band is the rapid multiplication of dislocations which you will visualize by polishing the sample surface to a highly reflective (i.e. smooth and mirror-like) state. As dislocations exit the surface, slip steps on the surface will roughen the surface, enabling you to actually observe the motion of the region undergoing dislocation slip first hand. By noting the nominal stress levels needed to create Lüder’s band slip in the regions of the stress concentration, you will be able to determine an apparent value for the stress concentration by comparing results with the straight gage section tensile bar. You will also measure the apparent angle of the Lüder’s band propagation with respect to the tensile axis by marking the angle using a Sharpy® marker, then measuring this angle later. Finally, with your last flat sample, you will perform a Rockwell B (100 kg over 1/16” ball) and Rockwell F (60 kg over 1/16” ball) hardness test to show the lateral scale of Lüder’s strain compared to the indentation size. Observe the Lüder’s bands that surround the indents, recording the image with a micrograph.

In a second portion of this experiment, you will gain first-hand knowledge of the effects of hydrogen embrittlement that affects steels with high yield stress, as well as a few other metals. Hydrogen enters the metal in the form of neutral hydrogen atoms, formed most often as a result of electrochemical “deposition” of positive hydrogen ions, H⁺ which combine with electrons in the metal to form H⁰. This is distinct from H₂, the diatomic gaseous form. Because the hydrogen can be driven in electrochemically by the creation of large surface concentrations of H⁰, the pressure of hydrogen dissolved interstitially in the metal can reach very high levels, exceeding the yield stress. In soft steels, this can be sufficient to precipitate gas bubbles that deform the metal known as blisters. In high strength steels, the hydrogen remains dissolved and resides as a positive ion between the positive metal cations. Its electron is part of the sea of electrons of the metal. It has a very high affinity for electrons and disturbs the bonding in the metal. The detailed mechanisms of embrittlement are not entirely understood but there are some researchers who believe the hydrogen assists in breaking metallic bonds while others believe that the hydrogen makes dislocation motion so easy that the material literally slides apart. In either case, the measured ductility of the metal is greatly reduced, and hence it is called hydrogen embrittlement. As it turns out, plating of metals such as chrome plating in acidic solutions is an ideal place to charge hydrogen into high strength metals such as
fasteners. Manufacturers of fasteners routinely bake out the hydrogen by heating the fasteners at about 200°C to allow the hydrogen to escape. If this is done right after plating, no damage is left behind but if the hydrogen is allowed to stay in the metal, it can precipitate creating permanent damage in the metal which cannot be removed by baking. Hydrogen embrittlement can occur in any situation where hydrogen is formed or exposed to the surface of metal. This can even include exposure to hydrogen gas. Thus it is important to avoid using high strength steels in contact with hydrogen.

In this lab, you will expose high strength spring steel to an acid electrolyte (Classic Coke®, pH ~ 2.5) and apply voltage from a 1.5 volt “C” sized battery for one hour, monitoring and recording the voltage and current. Note the capacity of the battery and report what fraction of the capacity was used during this experiment. You will measure the fracture stress of an untreated sample and the two samples that acted as the anode and cathode. Recall the anode is where the electrons are produced and cathode is where the electrons are used. Simultaneously, you will couple a similar sized sample of Mg alloy AZ31B (3% Aluminum, 1% Zinc, Balance Mg) via a wire as if these alloys had been bolted together, then expose them to the acidic electrolyte for one hour, monitoring current and the open circuit voltage developed electrochemically as the two different metals ACT AS A BATTERY. Testing the sample later will demonstrate hydrogen effects caused by mixing metals in a design. Take note bubbles you observe in the solution, do both electrodes bubble? Any other changes?
Detailed Experimental Procedure

Sample Preparation – This is to be done before starting the lab – Prep time approximately 1 hour

Each group will receive a stainless steel foil with 6 1010 alloy Steel samples (two tensile specimen and 4 samples with a center hole) that have been annealed in a furnace at either 700 °C or 900 °C for 1 hour and furnace cooled. Before going into the furnace the samples, foil and wooden toothpicks were weighed then wrapped in the foil and a total weight was taken. Each group will make a total weight measurement and then unwrap the samples and weigh the samples and the foil. The wooden toothpicks were added with the samples to reduce oxygen in the package and limit the amount of oxidation of the samples as well as to provide a carbon-rich environment to limit decarburization. The original weights will be given to you. A table is provided in this document for you to record your data.

After weighing, use double-sided tape to hold your samples to an Aluminum block for polishing. Wet abrade the samples starting with 240 grit SiC paper and rotating your block 90 degrees continuing on to the next grit paper in sequence from 320, 400 and 600 grit papers. The samples should be ground on each paper until the damage from the previous grit is removed.

The next step is to polish the samples. Rinse your sample in the sink with running water. The first polishing wheel by the door is used for the first polishing step. Wet the polishing cloth and then squirt 1.0 micron Alumina powder on the wheel. Turn the motor on high and hold your sample block on the cloth. Do this until your scratches are no longer visible. Rinse your samples in the sink and go to the last wheel next to the sink. Wet the polishing cloth, then squirt the 0.05 micron Alumina powder on the wheel. Turn the motor on high and finish polish your samples for a couple of minutes. Rinse your samples in the sink and blow dry with the hand dryer on the wall. Use a black Sharpie® marker to label your samples.

Your samples will be kept in Chris Pratt’s office until your scheduled lab time for testing.

The testing portion of the lab is conducted by two groups of three students each who will share their data.
Hydrogen Charging of High Carbon Spring Steel

You will be given 4 samples of Spring Steel and a Magnesium sample plus a bottle of Coke. There are two set ups for this part of the lab. Each group will be responsible for one set up, but will share the data at the end of the lab.

Set Up #1
Place 2 Spring Steel samples in a beaker through the plastic grid provided. (Mark the samples as positive or negative with a Sharpie.) Set up a circuit using a 1.5V battery with the positive side attached to one sample and the negative side attached to the other sample. Hook up a digital multimeter in series reading current in mA on the positive side and another digital multimeter hooked up around the battery to read voltage in Volts. One group member record time, Voltage and Current at 2 minute intervals for 1 hour, this data will be plotted in your results. Start collecting data once approximately 250ml of Coke is added to the beaker. After the hour is up, take the samples out and dry them off. Put the sample used as the anode in the grips of the MTS and run the machine at 0.1 inches per minute in the up direction. Record the maximum load for the sample. Put the cathode sample in the grips of the MTS, run the machine at the same rate and record the maximum load.

Set Up #2
Place a Spring Steel sample and a Magnesium sample in a beaker through the plastic grid provided. Connect one digital multimeter set to read current in mA with the positive lead attached to the Spring Steel and the negative lead attached to the Magnesium. Add another digital multimeter to measure voltage with the negative lead connected to the Magnesium side and the positive clipped to the sample side. To read the voltage, disconnect the lead from the sample and touch the positive lead from the multimeter. Record time, voltage and current at approximately 2 minute intervals for 1 hour once you have poured 250ml of Coke into the beaker. After 1 hour, take the samples out and dry them off. Put the Spring Steel sample in the grips of the MTS and run the machine at 0.1 inches per minute in the up direction. Record the maximum load for the sample. Also test the AS RECEIVED Spring Steel sample at the same rate and record the maximum load.

*While taking data of your voltage and current, move on to testing your annealed 1010 Steel samples.*

Observe the geometry of the fracture surface. It should look like a chisel. The two pieces should fit back together suggesting very little plastic deformation during the fracture. Does the break at the stress concentrator start off perpendicular to the local tensile stress? Does the fracture surface have a characteristic angle or flip from side to side?

Observations of Luders Bands
Each group should measure the dimensions of your annealed 1010 Steel samples. Record the thickness and width of the specimens. Insert the tensile specimen in the grips of the MTS machine. The crosshead will run at a rate of 0.1 inches per minute in the up direction until fracture. Watch the specimen while it is being tested. You will see Lüder’s Bands in your polished surface. The data will be saved to a text file and you can create a graph later with Excel or MatLab.

The sample with the center hole will be tested in the MTS at a crosshead rate of 0.01 inches per minute or 10x slower than the tensile test. While the test is running observe the area around the hole for the start of Lüder’s Bands and record the load. Continue the test until the Lüder’s Bands are on both sides of the hole and stop the test. Repeat the same tests with the other set of annealed 1010 Steel samples that have been annealed at a different temperature. All data will be shared with both groups.

### Weights of Annealed Sample

#### 700C Samples

<table>
<thead>
<tr>
<th>Samples</th>
<th>Weight before Annealing</th>
<th>Weight After Annealing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless Steel Foil</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Wooden Toothpicks</td>
<td></td>
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<tr>
<td>Total</td>
<td></td>
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</tbody>
</table>

#### 900C Samples

<table>
<thead>
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<th>Weight After Annealing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless Steel Foil</td>
<td></td>
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<tr>
<td>Total</td>
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Both groups of three who worked together during this experiment must prepare a document that includes all the data from their collaborative work, which is included as an appendix called Raw data for Lab#1, with the names of all 6 members of the combined group. **Hand this appendix in when you have completed it. Each group should have an electronic copy of this Raw Data.** This represents the shared information in your group from which each of the two teams of three will write their lab report. Concepts and ideas that you develop in the group of six should be included in this appendix.

Each group of three should then write up a lab report separately as a team of three, using the data and creating any additional analytics as needed. Each student in the group of three will receive the same grade. You may divide the work of writing the report to produce the best outcome. The goal of the lab report is to explain and document the phenomena you have investigated and observed, including background discussion of the phenomena. Describe how the knowledge you gained from this lab might be useful to design engineers and manufacturing engineers.