FIB and TEM Analysis of Nanoscale Thin Films

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Abstract:

Wire-drawn pearlitic steel has a refined lamellar microstructure of alternating ferrite (α-Fe) and cementite (Fe_3C) phases. Consequently, modeling the system by multilayer deposition and observing changes in periodicity and composition may reveal different applications. Using sputter deposition, thin film samples of alternating iron and carbon layers (with 0.1-5 nm periodicity) have been made to duplicate the microstructure.

In this study, we will use the dual-beam Focused Ion Beam (FIB)/Scanning Electron Microscope (SEM) to prepare cross-section Transmission Electron Microscope (TEM) samples. These will be characterized thoroughly, with the eventual goal of examining them *in situ* to view the behavior of the system during the annealing process. This will illuminate how small changes in Fe/C thin film compositions can result in immense differences in structure and hence, the properties.

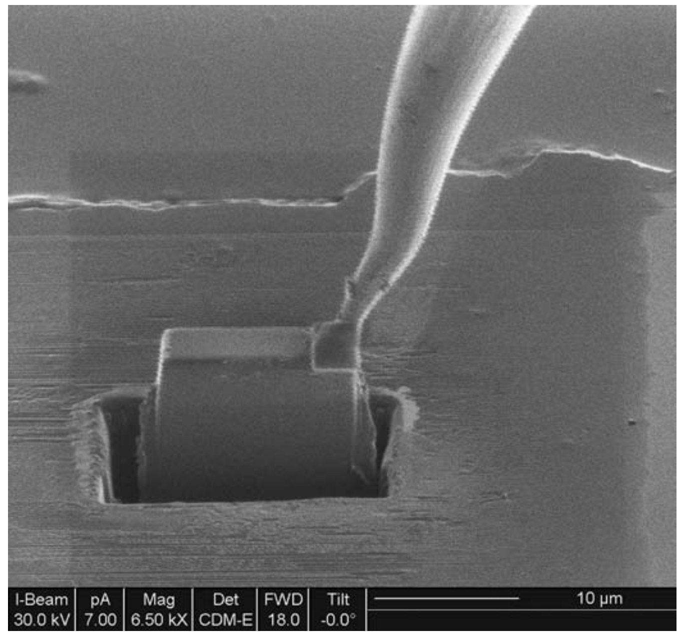

Figure 2: Transporting sample with Omniprobe.

Introduction:

Historically important in industry, pearlitic steel drawn to wire form has demonstrated a very high tensile strength without sacrificing its ductility and durability. Therefore, we have decided to reproduce this structure in thin film form to explore its properties at the nanoscale.

Since it has a lamellar microstructure of alternating ferrite (α-Fe) and cementite (Fe_3C) phases, we have modeled this system by sputter deposition, creating thin films with repeated layers of iron and carbon [1]. Previous research has suggested that as the drawing strain increases, the microstructure becomes finer, thus improving its strength [2]. Also, the size, shape, and composition of the carbide layers have a great deal of influence on the mechanical properties of steel [3]. Consequently, one main goal of our research is to vary the thickness of the iron-carbon layers to mimic the effect of fine lamellar microstructure on steel properties.

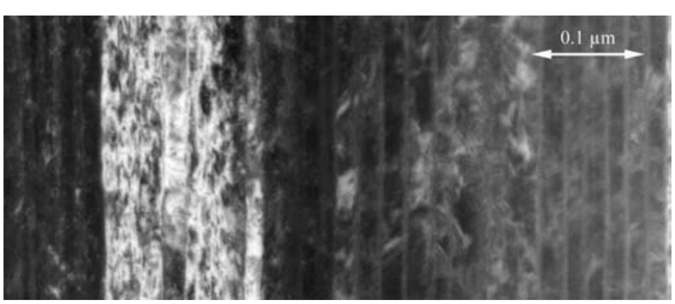

Figure 1: Bright field TEM of 0.2 mm pearlitic steel wire.

Because we want to observe the interactions between the layers, the most important viewing angle is through the cross-section. Although prior conventional sample preparation techniques yielded some results, the success rate was very low. Furthermore, the differential etching rate of the constituent elements created samples where bright-field imaging was possible, but quality high-resolution imaging was not. Therefore, using the FIB/SEM to shape and polish samples to uniform electron transparency is expected to produce higher quality samples at a better rate of success.

Another important aspect of this research is to examine the structural changes of the iron-carbon thin films during annealing. *In situ* TEM is a prime technique to record the behavior of the system during this process.

Experimental Procedures:

Sputtered thin film samples of alternating iron and carbon layers with 0.1-5 nm periodicity were provided. For this experiment, we examined a sample with layer periodicities 19.75 Å Fe/2.6 Å C, sequentially deposited to 3 μm thickness, starting with Fe and ending with C, on Si substrates with native oxide. 30kV Ga^+ ions in the FIB were used for rough shaping of the TEM cross-sections; they were finished using 10kV ions. These samples were then characterized using various TEM imaging techniques. We also planned to use *in situ* TEM to view the structural changes and reactions of the iron-carbon thin films during the annealing process in the temperature range of 350-450°C.

Results and Discussions:

Since we are interested in viewing the full thickness of the thin film, we used two methods to protect the area under examination from being etched during milling. The first approach was to deposit a rectangular 1 μm thick layer of Pt over the area we wanted to preserve. However, possible implantation effects produced negative results that prompted us to make a second sample using a marker to lay down a coat of ink instead. We then milled out a rectangular wedge with approximate dimensions of 14 x 10 x 4 μm using the FIB. The wedge was transported to a copper TEM half-grid using the FIB Omniprobe. Pt was deposited to weld the sample to the Omniprobe needle and, later, to the grid. Then, the needle was severed using the ion beam. Next, we thinned the wedge to below 0.1 μm with the FIB for electron transparency in the TEM.

At this point in the research, we have successfully used the FIB to mill out two sample wedges which we examined under the TEM. Upon inspection, we found that we could not differentiate the iron and carbon layers reliably, yet we could see the Si lattice image of the substrate. Examination of the first sample led us to believe that it was not yet electron transparent. However, after making another sample, we concluded that the iron and carbon layers may have reacted before being put into the TEM. Thus, these samples were not appropriate for use in the *in situ* phase of the research.

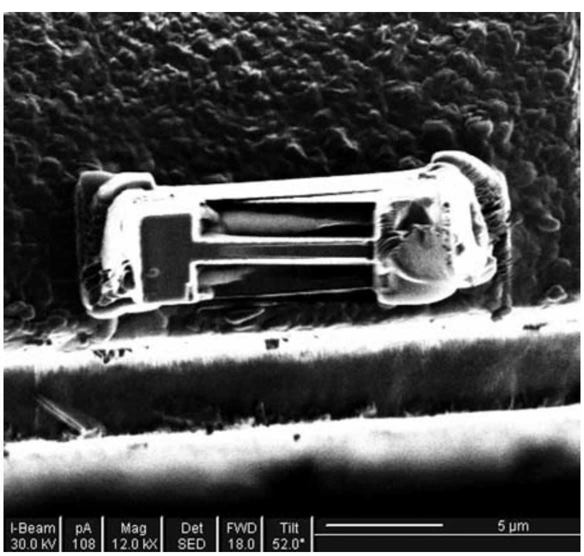

Figure 3: Thinning sample with FIB.

Conclusions and Future Work:

Because the sample seems to have reacted before conducting the *in situ* TEM, we were not be able to view the reactions and behavior of the iron-carbon system during the heating process. We suspected that the sample prematurely reacted during the FIB process or there were possible problems stemming from the sputter deposition of this particular geometry.

In the future, if we can prevent the problem of the sample reacting too early, then we will be able to conduct the subsequent phases of characterizing the thin films with the TEM.

Acknowledgments:

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References:

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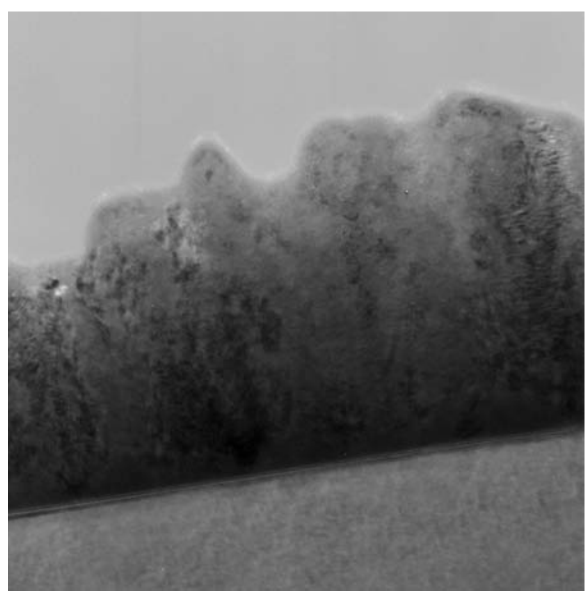

Figure 4: Bright field TEM of unsuccessful sample with iron/carbon grains on top of silicon.