Specimen Preparation

ENGR45 – Materials Science Laboratory

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Submitted: May 05th 2013

Abstract

Important information about a material and its physical properties can be determined by observing its characteristics under a high powered microscope. For the observer to be able to discern the qualities of the metallurgical microstructure for a material a sample must be carefully prepared in such a way that its characteristics will be properly translated through the magnifcation process. In these samples surface flaws such as grooves, dents and cracks must be removed so that only the natural properties of the material remain which are then made apparent through magnification. Proper specimen preparation provides a means to gradually eliminate these distortions so that only the true characteristics of the materials being studied remain. The visualization of the metallurgical microstructure—such as material grain size and grain boundaries—is then made clearly apparent through the image produced by an optical (or other) microscope. In this experiment, a sectioned 10mm by 10mm sample of A36 steel was prepared so that it could be studied under an optical microscope capable of 400x magnification.

Procedure

A pre-sectioned 10mm by 10mm specimen of A36 steel was selected to be mounted within a puck for preparation. The surface of the material was cleared of any residual dust or grime by lightly tapping it with a hammer against a hard surface and then wiping clean with a lint free cloth. It was then inserted into the bottom of mold cylinder with the mold base already outfitted to the device. The flattest and cleanest surface of the A36 sample was carefully aligned so that it would remain in the center of and surrounded by molding for the final mounting puck. Approximately 40ml of phenolic powder—a black and finely grained substance—was slowly added to mold cylinder. The powder was then tamped to be nearly flat and the mold ram was then inserted into the top to contain the sample and the powder, being careful not to disrupt the position of the sample and invite powder to creep under its bottom surface.

To finalize the mounting puck, the mold cylinder was placed within a Buehler brand specimen mount press machine where a heat between the range of 100°C to 130°C was applied under high pressue for 18 minutes. Before leaving the puck to bake for this duration of time the max pressure of about 4200 psi was applied for a few seconds (with the applied heat) and was then quickly removed; this allowed the

"burping" of the cylinder so that trapped gas could be allowed to escape from the mold cylinder. Reapplication of the same pressure for the duration of the baking of the puck required monitoring and occasional adjustment ever two or three minutes. This is because the applied pressure gradually decreased over time, an issue that is likely attributable to the poor maintenance of the equipment. The maximum temperature achieved during the process was 130.1°C. A picture of the apparatus is shown in **Figure 1** on this page.

Figure 1. Specimen mount press.

The heating element was turned off and the mold cylinder was removed from the mount press machine to cool after 18 minutes of baking. After about three minutes of cooling vertical pressure was applied to the top of the mold cylinder to remove the mold base, mounted specimen puck and mold ram. An unexpected complication occurred at this point: while the mold base and puck were successfully removed the mold ram became jammed within the cylinder. Operator error led to too much pressure being applied which resulted in the bending and breaking of the specimen mount press machine. This problem was likely attributable to an incorrectly prepared specimen and/or poor handling of the phenolic powder and equipment. The A36 steel specimen was almost entirely to one side of the puck and a large amount of powder had found its way under the exposed surface of the sample. Because of the breaking of the machine a second replacement sample could not be prepared and previously mounted samples were used for portions of the specimen preparation procedure relating to the grinding, polishing and etching of the sample.

The removal of surface defects was performed first through three separate grinding steps using 120, 240 and 320 grit sandpapers, and then through two fine grinding/polishing steps using velvet and nylon cloths. At the conclusion of grinding and polishing the sample was etched with 5% nital solution which contained a mixture of 5% concentrated nitric acid in alcohol and 0.5% to 5.0% dilute nitric acid.

While grinding with 120 grit sandpaper the puck was pushed along the surface at the same angle without any rotation being allowed; this ensured that all grooves along the surface upon the completion of this step were parallel with one another. After five minutes the sandpaper was changed to 240 grit and the same procedure was repeated except that the puck was rotated 90°. This caused the new grooves created by the finer sandpaper to run perpendicular to the original grooves. In both of these steps small amounts of water were regularly sprayed on the sandpaper for lubrication to assist in the grinding process. The 320 grit sandpaper was attached to a mechanical wheel that was capable of rotating at high speeds. For this final grinding step water was sprayed on the surface of the sandpaper and the surface of the specimen puck was slowly glided at rotated from the edge of the wheel to its center. A picture of this device is shown in **Figure 2** on this page. This process was repeated until all of the parallel grooves from the prevoius step were removed which took about 10 minutes.

Figure 2. Grinding and polishing station.

For further grinding and polishing the above steps described for the 320 grit sandpaper were followed for two additional mechanical grinding wheel stations, the first outfitted with velvet cloth and lubricated with a solution of 1.0 micron alumina suspended in water and the second outfitted with nylon cloth and lubricated with 0.05 micron alumina suspended in water. The sample was polished for approximately 10 minutes at each station using the rotation method from the outside to the inside of the wheel as before. Upon completion the surface of the mounted A36 steel specimen was highly polished and reflective like a

mirror. To finalize the sample a q-tip soaked with the 5% nital solution was quickly swabbed over the surface of the specimen where it was left for only five seconds. It was then quickly rinsed with water followed by alcohol and then dried with a cloth. The resultant specimen was a highly polished surface with its luster removed by the etching process and no defects visible to the naked eye.

Results

Placing the prepared A36 steel sample under an optical microscope showed that the grinding and polishing procedure was successful in removing surface defects and bringing out the grain structure and grain boundaries of the material. Only a few noticeable residual defects remained. The results of this successfully prepared sample are shown in **Figures 3 through 6** on the following pages.

Conclusion

Procedural difficulties existed in earlier steps of this experiment resulting in a piece of equipment being damaged and subsequently disabled. Additional care must be exercised by the operators of these machines so that these kinds of problems do not occur again in the future. Although the specimen mount press had been broken, the following grinding, polishing and etching steps resulted in a very clean sample. The final result produced a polished steel sample where the properties of the material could be readily and clearly observed under a microscope without groove distortions and interferences.

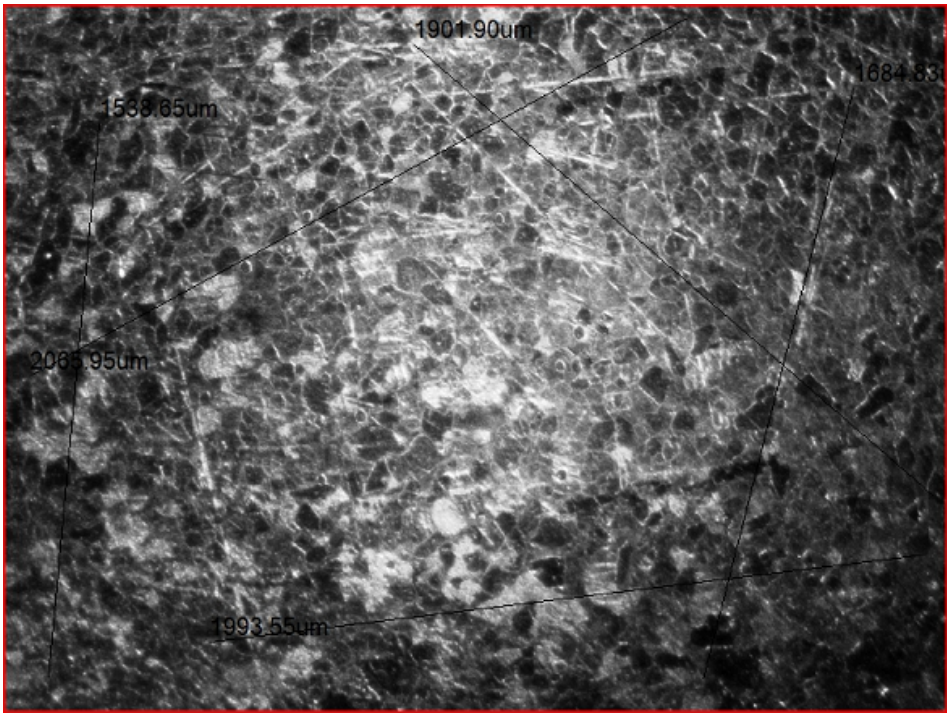

Figure 3. A36 steel sample at 40x magnification.

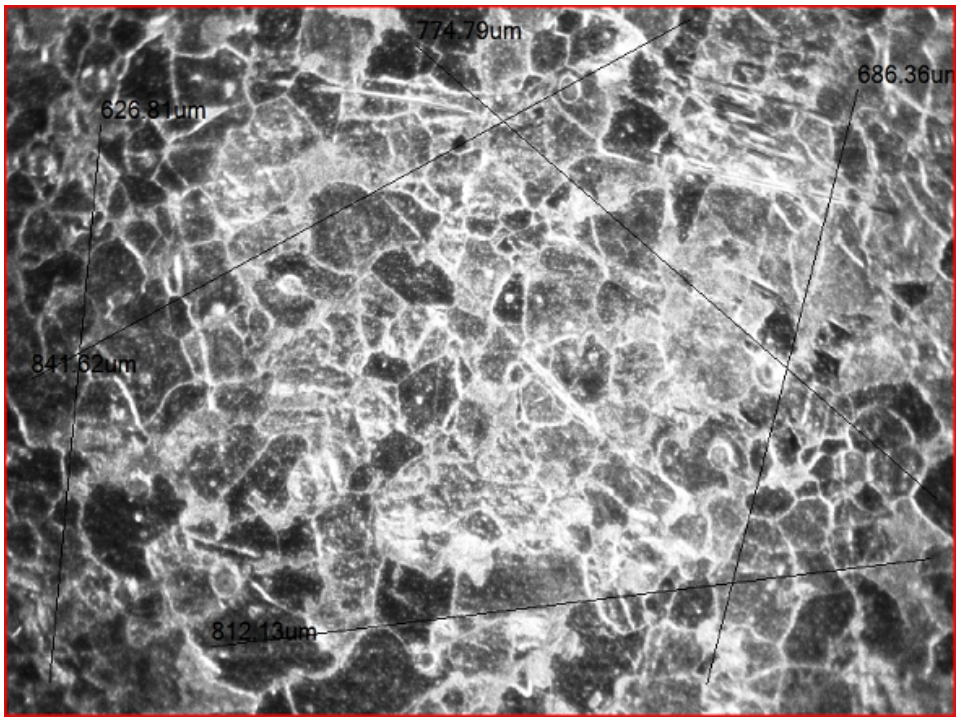

Figure 4. A36 steel sample at 100x magnification.

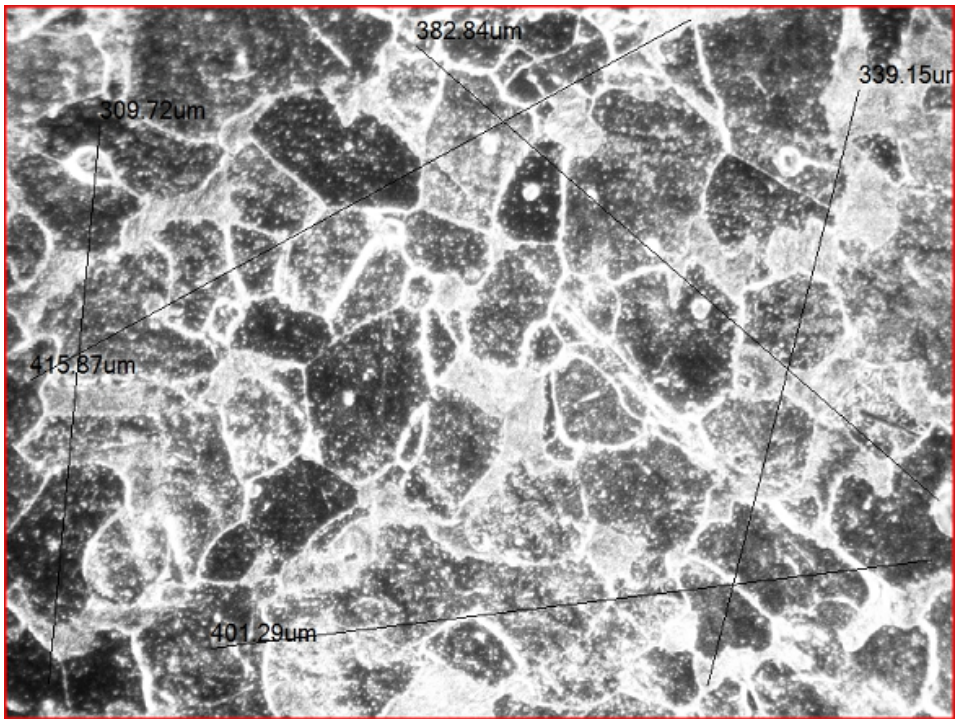

Figure 5. A36 steel sample at 200x magnification.

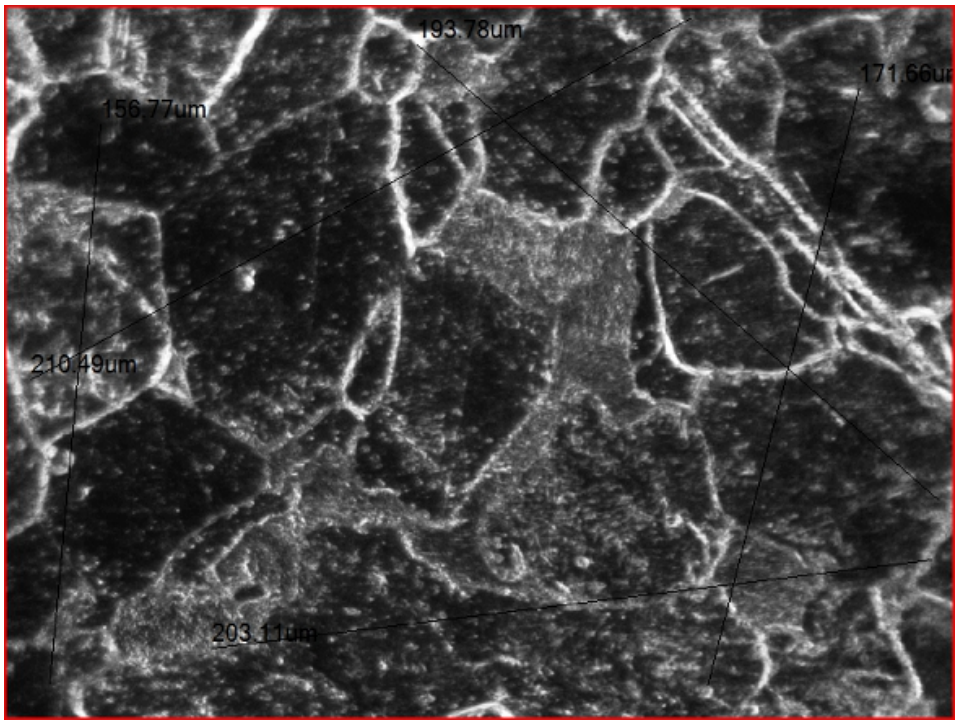

Figure 6. A36 steel sample at 400x magnification.