Microscopic Examination

ENGR45 – Materials Science Laboratory

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Abstract

Important properties of a material can be discovered or confirmed by observing its physical characteristics under a high powered microscope. These characteristics include grain size, variations in light reflections, presence of impurities, surface contaminations, defects, and a host of other microscopic details that are not accessible to the unaided eye. In particular, the careful study of the material's grains under a microscope can produce a great deal of information about its behaviors and qualities under various conditions, such as fluctuating temperatures and pressures, and its compositional characteristics after experiencing a treatment, such as undergoing a diffusive process. Three unique samples—A36 steel, a lead-tin solution, and a hardness calibration disk—were visualized under an optical microscope so that the grain size number could be determined for each. A value was obtained only for A36 steel due to the inability to obtain visual clarity on the grain boundaries for the lead-tin solution and hardness calibration disk samples.

Sample	Grain Size Number	Nominal Diameter	Avg. Grain Section Area
A36 Steel	6.59	~33µm	~1.15mm^2 10^{-3}

Procedure

Material samples were observed under a high power optical microscope capable of 40x, 100x, 200x, and 400x magnification: a mounted and specimen prepared A36 steel sample, a lead-tin solution sample, and a hardness calibration disk with hardness rating of 26.2 +/- HRC. Before loading any of these samples into the device time was spent to learn about how to correctly operate the microscope so that optimal visual results would be obtained. This required practicing how to perform the many kinds of adjustments to its dials and control settings in order to obtain a desired focus and resolution for an image. The microscope had a wide assortment of options to control a resolved image, from magnification filters to light intensity adjusting knobs (see **Appendix I** for more details regarding the features of the microscope used in this experiment).

A special camera attachment was on hand to digitally capture and record the observed images into a computer. This allowed for the same image from within the eyepiece to be loaded into a computer and projected onto a monitor, which aided in the process of investigating the material. The computer software that interfaced with the camera and microscope allowed for these images to be manipulated for an enhancement in the clarity of the material's magnified features. In combination with the visual controls on the microscope, a great deal of customization could be made to the recorded image: color, contrast, brightness and other visual characteristics could be adjusted from within the computer application while focus, light filter and light intensities could be adjusted on the microscope.

Each of the three samples was loaded individually onto the specimen table of the microscope. In each case, resolving the image of a sample involved selecting the intended lens magnification setting of either 4x, 10x, 20x, and 40x (which, in conjunction with the default 10x magnification, produced true magnifications of 40x, 100x, 200x, and 400x) and adjusting the lens's vertical position over the specimen so that an appropriate focus could be obtained. For samples with well prepared, smooth surfaces this allowed for the entire visible area to appear in focus. However, for samples with inconsistencies in surface height the focus had to be carefully adjusted so that the characteristics of the visual area could be obtained in stages (focusing on higher regions resulted in the blurring of the lower regions, while focusing on lower regions blurred the higher regions). In cases where a sample had a highly smoothed and polished surface one image was captured at each of the different magnification settings. For all other samples multiple images had to be captured at each magnification level to account for the lack of focus available through the entire visible area. The most relevant of the recorded images for the three samples can be seen in **Picture 1** through **Picture 8** located at the end of this report.

With the aid of the computer software, grain counting lines of measured lengths were drawn on each of the obtained images. The linear intercept procedure was used to measure the grain size number of specimens with clearly defined grain boundaries. The total number of grain boundary intercepts were counted along each line and then this number was divided by the length. An average was taken of the five values obtained from each of these lines and assigned to variable P_L. This value was then substituted into the following equation to solve for the grain size number:

$$n = -3.3 + 6.65 \log_{10}(P_L)$$

Results

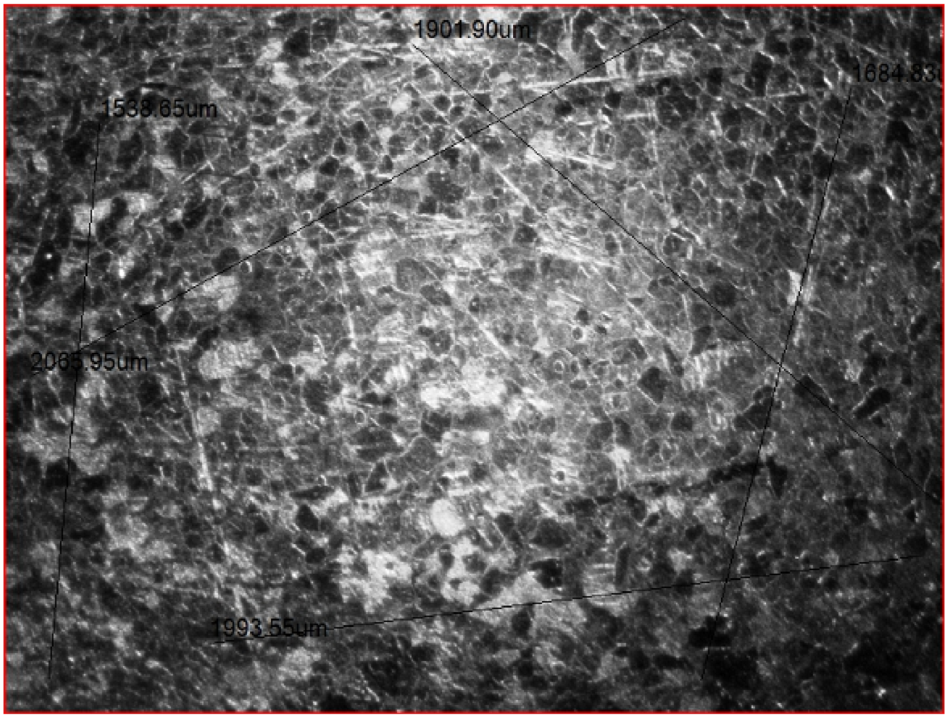
The sample used for A36 steel had been properly prepared beforehand so that all of its larger sized surface defects were removed, leaving a very clear representation of its true microscopic qualities. This preparation also allowed the microscope to easily obtain focus across the entire viewable area of the sample because it was of uniform height; this allowed for the grain size number of 6.59 to be determined using the linear intercept procedure mentioned above. This grain size number correlates to an average grain having a nominal diameter of ~33μm, intercept count per millimeter of approximately 33, area of average grain section ~1.15mm^2 10^{-3}, and grains per millimeter at 1x magnification of ~900. Noticeable visual properties of the A36 steel sample include: 1) variations in grain color from light to dark with many shades of grey; 2) residual scratches and markings that were not removed during the specimen preparation procedure; and 3) small circular impurities existed in the centers of some grains. These results can be seen in **Picture 1** through **Picture 4**.

The lead-tin solution had not been prepared properly and because of this the surface was rigid and had many variations in the surface height. This produced difficulties in obtaining a correct focus on the entire visual area of the sample—only the 40x magnification setting produced a clear image of the material (see **Picture 5**). Grain boundaries were not well defined overall and because of this a grain size number could not be determined. Noticeable characteristics of the lead-tin solution include the appearance of only two colors for the material's grains, light grey and dark grey, and the presence of dark, curved markings at various locations at the surface.

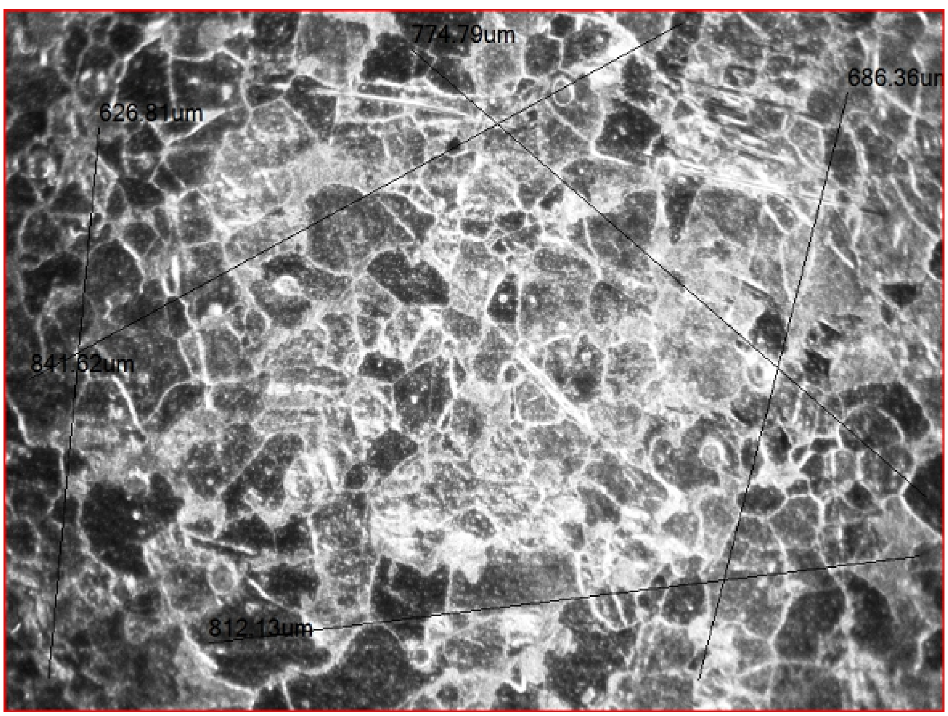
The hardness calibration disk was unique in that its grains could not be distinguished, even under 400x magnification. For this sample a hardness test indentation was magnified along with its surrounding area which allowed for unique observations to be made about a clearly defined and well pronounced surface defect. The most noticeable attribute is the dark ring surrounding the indentation, which is a result of the plastic deformation experienced in that location of the sample (see **Picture 6**). Increased magnification inside of the indentation shows a mostly smooth surface with areas where graininess and variations in height appear. This suggests that perhaps with a higher magnification in this region the grains of the material would become visible. Unfortunately, because this region occurs on a sloping surface, difficulties in focusing the lens prevented any further inquiries to be made into the response of the material to a hardness testing indentation. It is, however, expected that the material will show unique characteristics for its plastically deformed regions when compared to its non-plastically deformed regions.

Conclusion

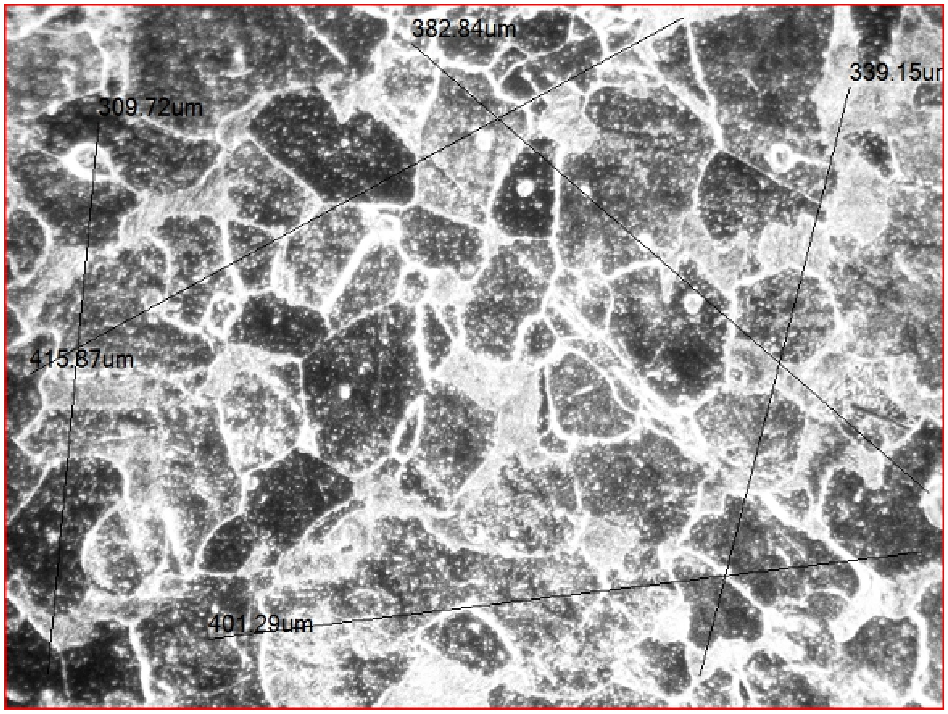
Perhaps the most important and telling observation made during this entire experiment is that a properly prepared specimen is absolutely necessary for effective qualitative analysis to be performed using a high power microscope. A sample must be rid of its surface defects and also have a leveled examination plane so that a magnified image includes details for the entire viewable area after the lens has been focused. The A36 steel sample was the only one of the three specimens that was properly prepared. Subsequently, it was the only material for which a grain size number could be determined.



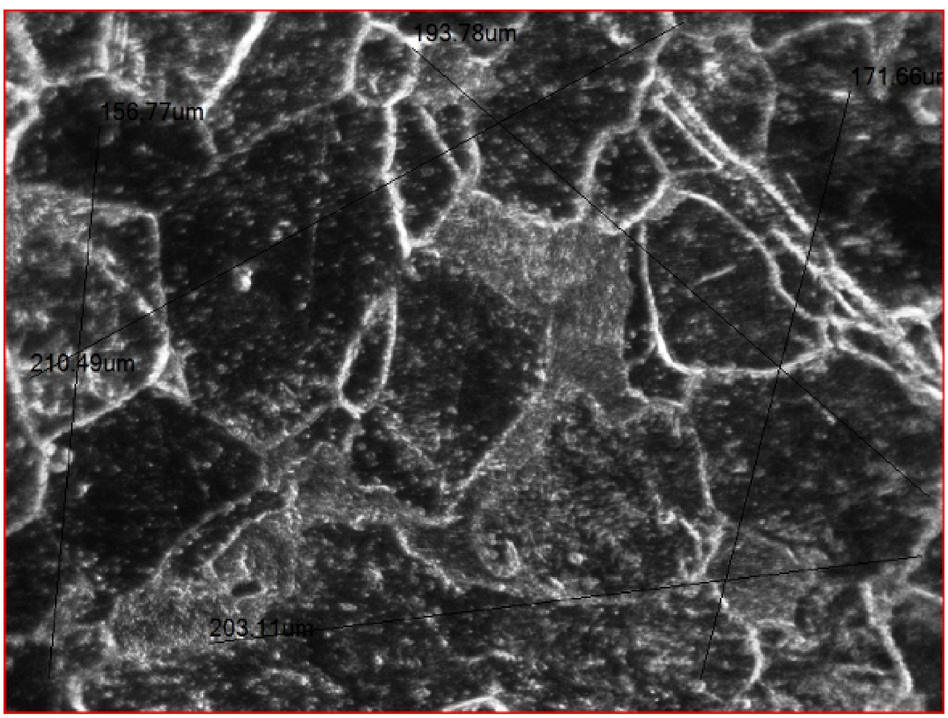
Picture 1. A36 steel sample at 40x magnification.



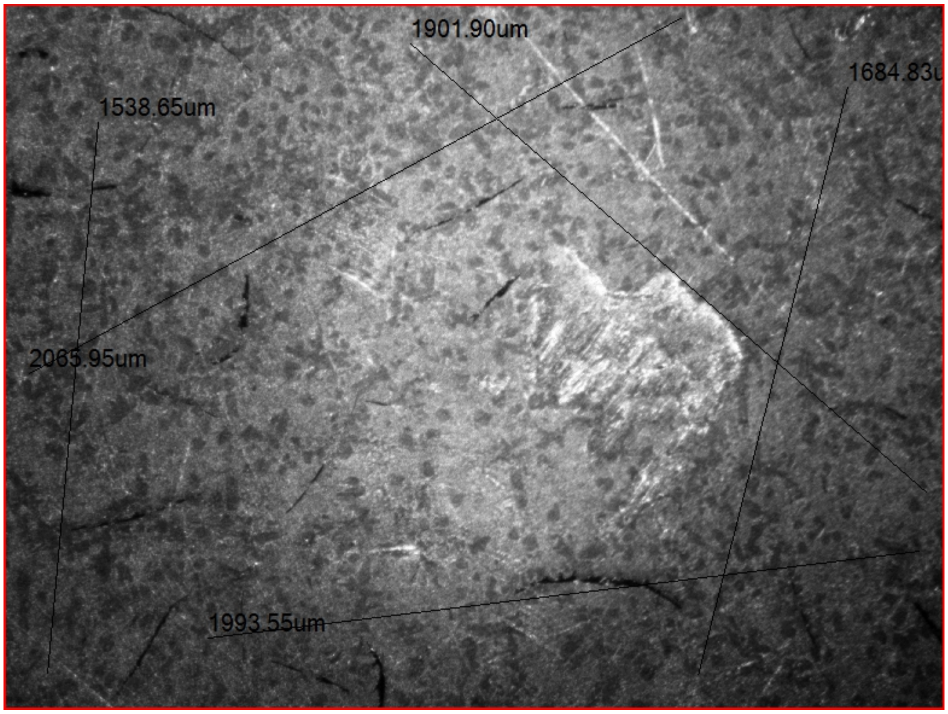
Picture 2. A36 steel sample at 100x magnification.



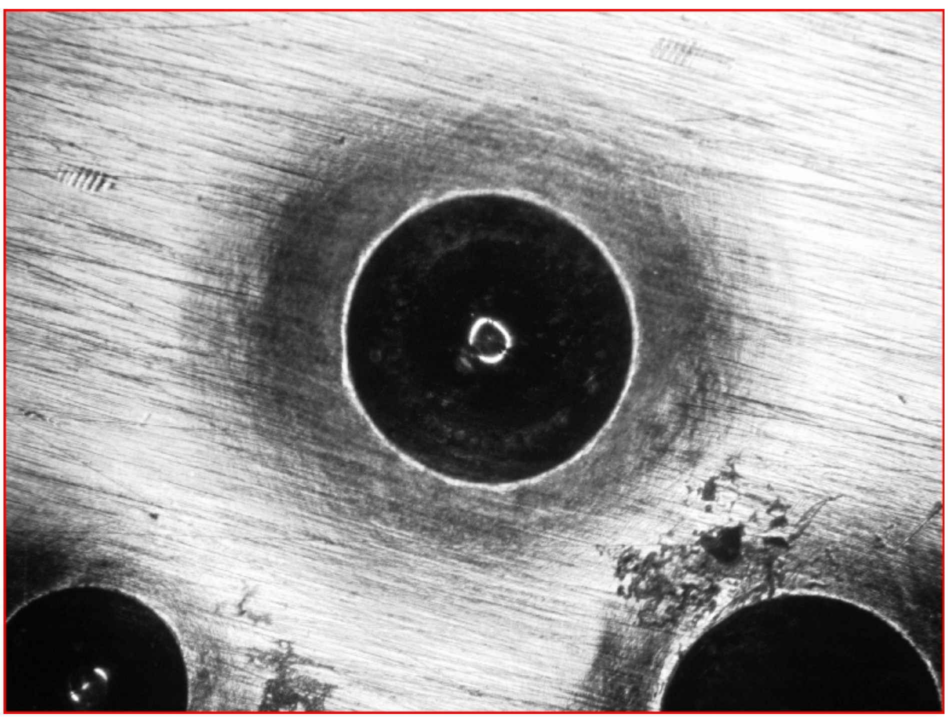
Picture 3. A36 steel sample at 200x magnification.

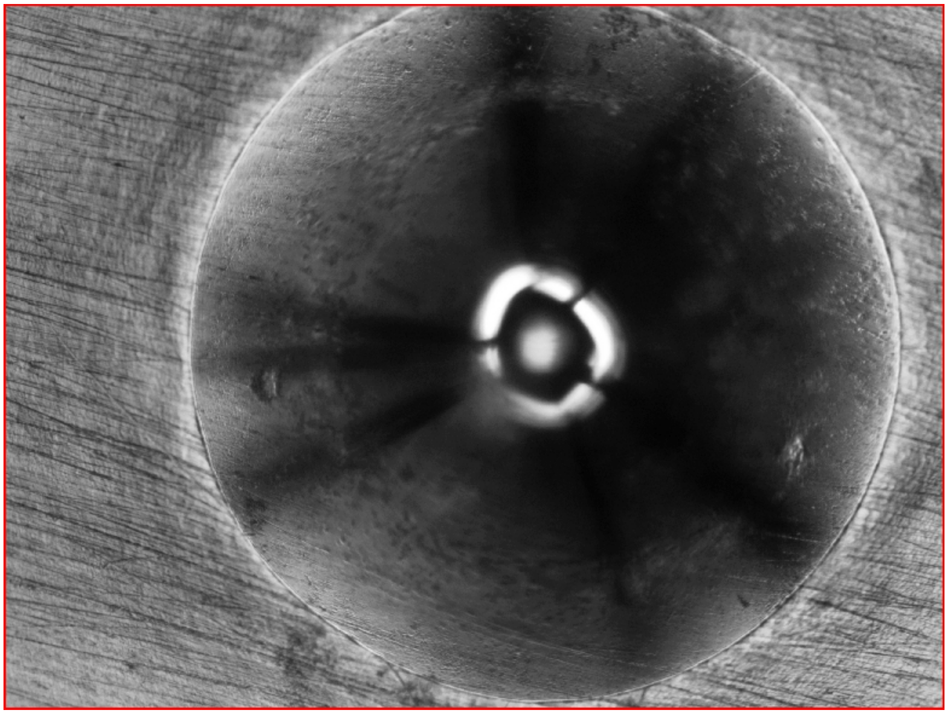


Picture 4. A36 steel sample at 400x magnification.

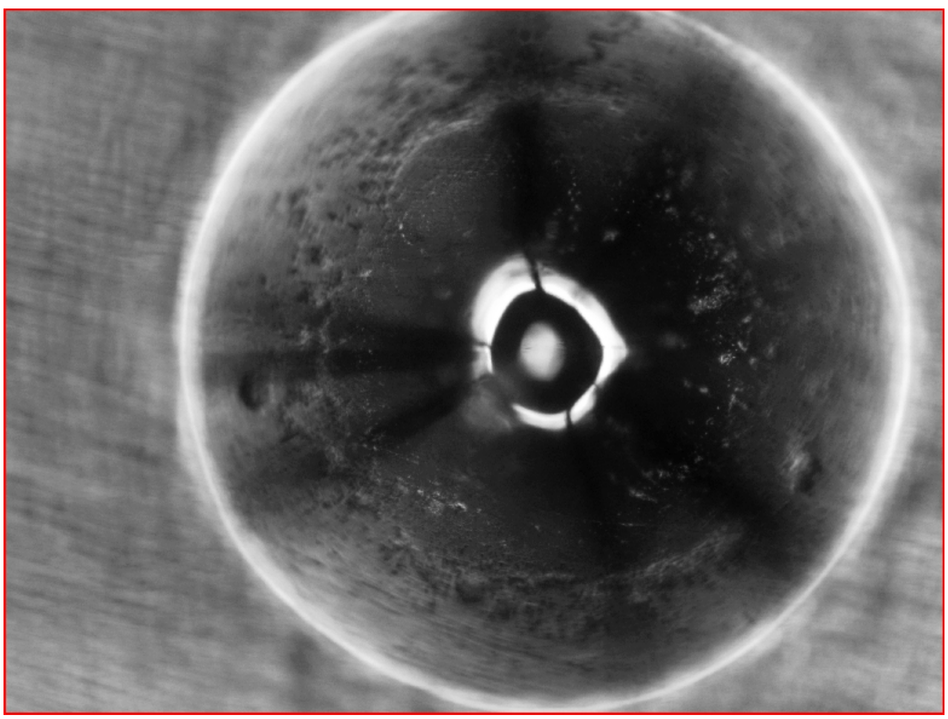


Picture 5. Lead-tin sample at 40x magnification.


Picture 6. Hardness calibration disk at 40x magnification.



Picture 7. Hardness calibration disk at 100x magnification.



Picture 8. Hardness calibration disk at 100x magnification (adjusted focus).