Washington University in St. Louis

MEMS 205 Mechanics and Materials Science Laboratory

Lab 3: Microstructure

Washington University in St. Louis

JAMES MCKELVEY SCHOOL OF ENGINEERING

Section: Group D (Tuesday 12 PM)

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Experiment Date: Tuesday 2/18/2020

Report Submission Date: Tuesday, 2/25/2020

We hereby certify that the lab report herein is our original academic work, completed in accordance with the McKelvey School of Engineering and Undergraduate Student academic integrity policies, and submitted to fulfill the requirements of this assignment.

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Abstract

We determined the grain size and shape by using an optical microscope to observe the microstructure of 1005, 1018, 1045 steel and copper. The process consisted of mounting, grinding, polishing and etching each specimen. This was to ensure a flat, smooth and mirror-like surface so that a clear picture was seen under the microscope. Images of the 1005, 1018, 1045 steel and copper were obtained from the microscope. To determine the grain size we used the linear intercept method and counted the number of intersections of grains on a line that was drawn on the images. Once the number of intersections was obtained we divided the length of the line by the number of intersections to obtain the average grain size of the materials. From our observations we determined that the grain size of the steels were smaller as the carbon concentration increased. We also noticed that when you quench steel, nucleation dominates and grains are not allowed to grow. In addition, both the copper and the steel went through heat treatment processes. In heat treatment recrystallization causes the formation of new grains and thus, decreases the average grain size. As a result, we see small fine particles in the 1045 steel and more needle shaped grains in the 1018 steel which is characteristic of the martensite phase. For the copper we determined that the grain size of the specimen was 4.8 μm. While our average grain size for 1018 and 1045 steel was 1.7 and 1.2 μm respectively.

Introduction

Spartan Light Metal Products is looking to determine the average grain size and shape for several metal specimens created by their Research and Development team. The microstructure of a metal is directly related to its mechanical properties [1]. To gain some insight into the mechanical behavior of these created metals, we examine and compare the microstructure of two created metal samples from Spartan to three other metal samples. The three other samples consist of a single copper specimen that has been annealed, cold worked, and tempered and two steel specimens that have been austenitized, quenched, and tempered.

The processing of metal affects its grain size. Cold working increases grain size on the cold-rolled face. Annealing causes nucleation. Nucleation occurs at grain boundaries, reducing the average grain boundary size as nucleation draws mass from pre-existing grains to create new grains. Quenched austinite forms martensite, which has needle-like grains [1]. As with annealing, when martensite is tempered, nucleation begins, causing new grains to pull matter from old grains, thereby reducing the average grain size of the material. With all other variables held constant, materials with a small grain size will be stronger than materials with a large grain size.

A metal's microstructure can be observed through a microscope by applying a multistep process that begins with sectioning and mounting, then followed by grinding and polishing, and is completed with etching. In sectioning and mounting, a sample is trimmed to an easily manipulated size and then set in a cement cylinder for ease of handling during the grinding and polishing steps. Microscopes have a very small depth of focus [2], so in order to obtain a clear image of the grain structure, the outer surface of the sample must be almost perfectly flat. The grinding and polishing steps are used to create this flat face as the specimen is ground with progressively finer sandpaper and then polished with an abrasive of alumina particle solution of different concentrations. This alumina solution is much finer than sandpaper, allowing the face of the specimen to be smooth and mirror-like. The grain boundaries are revealed by etching, where each specimen is treated with a solution that undergoes a chemical reaction with the specimen. Grain boundaries are more chemically active than the rest of the specimen, so the chemical reaction will cause the most change at the grain boundaries [3], making the grain boundaries visible under a microscope.

Grain size, an integral component of microstructure, can be determined by inspection. There is more than one way to determine grain size by inspection, but we use the linear intercept method [4] since it is intuitive and can be performed without highly specialized equipment. The average grain diameter, or grain size, is the average lineal intercept, which is defined as the average number of grain boundaries crossed by a line of unit length; described by:

$$\ell = \frac{L_T}{P}. \tag{1}$$

ℓ is the average lineal intercept, $P$is the number of grain boundaries crossed by L_T, which is a line of some length. Line $L_T$is drawn on a picture of the microstructure taken through a microscope. $L_T$is normally in the µm range.

Experimental Methods

To examine the microstructure of each specimen, we used an optical microscope. First, we put a specimen into the bottom of a cup, if the specimen was too big to fit into the cup we trimmed it until it could lay flat in the cup. Then we mixed 5 mL of the Koldmount liquid with 10 mL of the Koldmount powder, maintaining this 2:1 ratio of powder to liquid. This was mixed for at least 30 seconds, to ensure a consistent mixture, then poured over each specimen so that it was submerged and about ¾ of the cup was filled with the liquid-powder mixture. The cups were then left to harden under the hood for a week.

The excess hardened Koldmount was ground off using a course grinder to expose the surface of the material. The next step was to grind the exposed face of the specimen on a grinding wheel at varying grits. The grinding wheel was set to rotate between 200-225 rpm, with 180 grit carbide paper. Water was set to flow at a constant stream onto the sandpaper while applying moderate pressure to the specimen. During this step, the specimen was checked periodically to see if there were line scratches all in one direction on the surface of the metal (Fig. 1). In this step, we made sure to keep the orientation consistent. After the line scratches looked parallel the specimen was rinsed with water and dried.

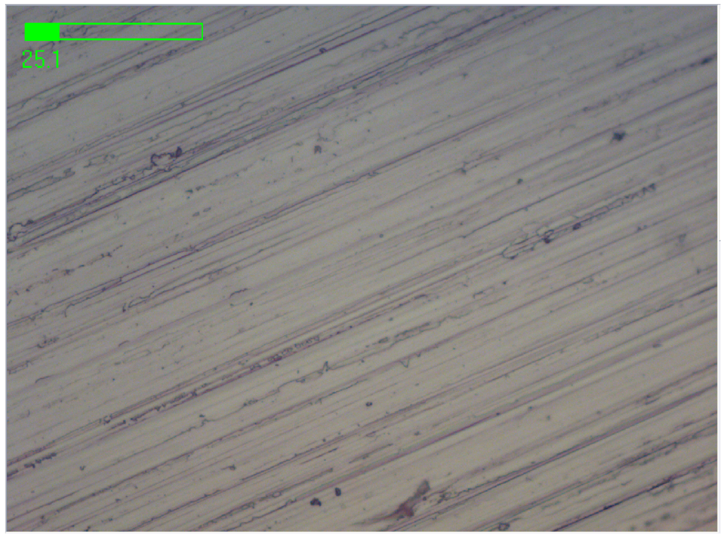

Fig. 1 Copper specimen microscopic photo after a grinding stage, showing the surface scratches being uniformly parallel and in one cohesive direction.

The specimen was then rotated 90˚ and ground using 320 grit paper until only scratches showed in this new orientation. Lastly, the 600 grit was used with the repeated processes, making sure that the specimen was rotated 90˚ from the last grinding. Rotating the specimen ensured the surface was being scratched in a consistent direction for the varying grits. Table 1 shows the times used for each of the grinding steps based on the specimen.

Table 1 Grinding times for each specimen at varying sandpaper grits

Specimen	Sandpaper Grit and Time Spent Grinding		
	180	320	600
1018 Steel (Tempered for 30 minutes)	18 mins	5 mins	3.5 mins
1045 Steel (Tempered for 30 minutes)	20 mins	5 mins	3.5 mins
110 Copper(Tempered for 18 minutes)	5 min	5 mins	3.5 mins
110 Copper (Tempered for 4 minutes)	5 min	5 mins	3.5 mins

Next, we polished the surface of the specimen using multi-staged polishing processes with green velvet cloths. To polish the specimen we used 5 μm, 1 μm and 0.05 μm alumina particle solutions that were sprayed onto the surfaces of each specimen. Water was running at about one drop per second and the polisher was spinning between 150-200 rpm. The first polishing stage utilized the 5 μm alumina particle solution that was applied to the specimen. The specimen was held to the polisher with slight pressure. This was done until the sample had slight scratches going in one direction that was 90˚ compared to the scratches from the previous grinding stage. The sample was then rinsed with water and air-dried. This same process was repeated for the1 μm alumina particle solution making sure the specimen was rotated 90˚ again, ensuring the surface is nearly mirror-like at the end of this stage. The final polishing stage utilized the previous steps with the 0.05 μm alumina particle solution. The specimen was rotated while polishing during this stage, ensuring the final surface was smooth and shiny. The final polisher was run at 138 RPM, whereas the first polishing stage was done at 200 RPM and the middle stage was at ran at a minimum of 125 RPM. After the final polishing, the surface was rinsed with water as usual but added an alcohol rinse before the etching stage. Table 2 displays the time spent polishing each specimen for each polishing stage.

Table 2 Polishing times for each specimen at varying alumina particle solution compositions

Specimen	Alumina Particle Solution Compositions and Time Spent Polishing		
	5 μm	1 μm	0.05 μm
1018 Steel (Tempered for 30 minutes)	5	3	2
1045 Steel (Tempered for 30 minutes)	5	3	3
110 Copper (Tempered for 18 minutes)	5	2	2
110 Copper (Tempered for 4 minutes)	5	3	2

Each specimen was then etched under the hood using the proper etching solution for the copper and steel specimens. Table 3 shows the different etching solutions for each material. The specimen was first sprayed with alcohol. Then the appropriate etchant was then used on the specimens and after 25 seconds the sample was rinsed with water and air-dried in the hood.

Table 3 Etching solution used for each specified material

Etchant Solution	Material
Ammonia Hydrogen Peroxide(3%) DI Water	Copper
Ethanol Nitric Acid	Steel

Lastly, each specimen was taken to the optical microscope and placed on the stage with the surface of interest faced down, as depicted in Fig. 2. An image was then displayed and focused on the computer screen to capture a picture of the microstructure of the specimen to determine the average grain boundaries. The magnification was at GX41 50X for each picture.

Fig. 2 Laboratory optical microscope [5].

Table 4 lists the equipment utilized during the experiment with dedicated serial numbers, model numbers, and instrument makes. Table 5 describes the type of sandpaper and the grit of sandpaper that was used.

Table 4 List of equipment used to produce specimens to be analyzed by microscopy

Equipment	Serial number	Description	Uncertainty
Grinder/Polisher	3531	Model #: 825-300-300 Leco, Spectrum System 1000	-
Polisher	SS20176	Buehler, Ecomet II	-
Polisher	SS20176	Model #: 825-400-100 Leco, SS_200	-
Microscope	2D64202	Model #: GX41F Olympus Corporation	-

Table 5 Sandpaper of varying grits for grinding of the specimens

Grit	Type	Disc Diameter (in)	PROD LOT #	Jumbo Serial #
180	Silicon Carbide	8	-	-
320	Silicon Carbide	8	07W-04	20308
600	Silicon Carbide	8	07W-01	16803

Results and Discussion

The grain size of a material can have strong effects on the properties of a given material [1]. In addition, the microstructure of materials can be significantly influenced by the condition of material experiences. For example, annealing and then quenching a material, will not allow the grains of the material to grow, as a result, the microstructure will have a large number of small grains. Also, tempering or annealing without quenching will allow the grains to grow and increase in size as nucleation is activated at higher temperatures. Table 3 shows the results of inspecting the microscope photos and analyzing the number of grain boundary intersections for each randomly drawn line on the images. The variables are consistent with Eq. 1, with the final average grain intersect length calculated as ℓ. Table 6 represents the data analyzed for the microstructures of the specimens in Figs. 3 to 6.

Table 6 Final calculated average grain size, for various materials under different initial conditions.

Specimens	L_T (μm)	P	ℓ (μm)
110 Copper (Tempered for 18 minutes)	267.53	56	4.8
1005 Steel (Provided)	353.41	42	8.4
1045 Steel (Provided)	310.50	57	5.4
1018 Steel (Tempered for 30 minutes)	301.82	177	1.7
1045 Steel (Tempered for 30 minutes)	56.98	46	1.2

In Table 6, values were obtained using Eq. 1 and counting the number of grains intersecting the randomly drawn lines. Our data show that as carbon composition increases in steel, the microstructure grain sizes decreases. This trend is expected as the higher carbon steels are generally stronger than low carbon steel and grain size is an important factor in strength [1]. Small grain size can mean higher strength. For the 1018 and 1045 steel specimens, the grains are very small. As a result, it is difficult to get a precise count on the grains as they pass under the thicker line that was drawn on the microscope image. However, the average was performed in an attempt to reduce errors caused by miscounts.

Figure 3 shows an image of the microstructure of copper taken at 50 times magnification. Table 3 shows the average grain size of this specific material which is an average grain size of 4.8 μm. The expected grain size for pure copper is 60 μm [6]. However, the difference is, our sample was tempered for 18 minutes after cold rolling. During tempering recrystallization takes place and the grain will shrink as new grains are formed. Thus, it makes sense that we would observe a smaller grain size than pure copper.

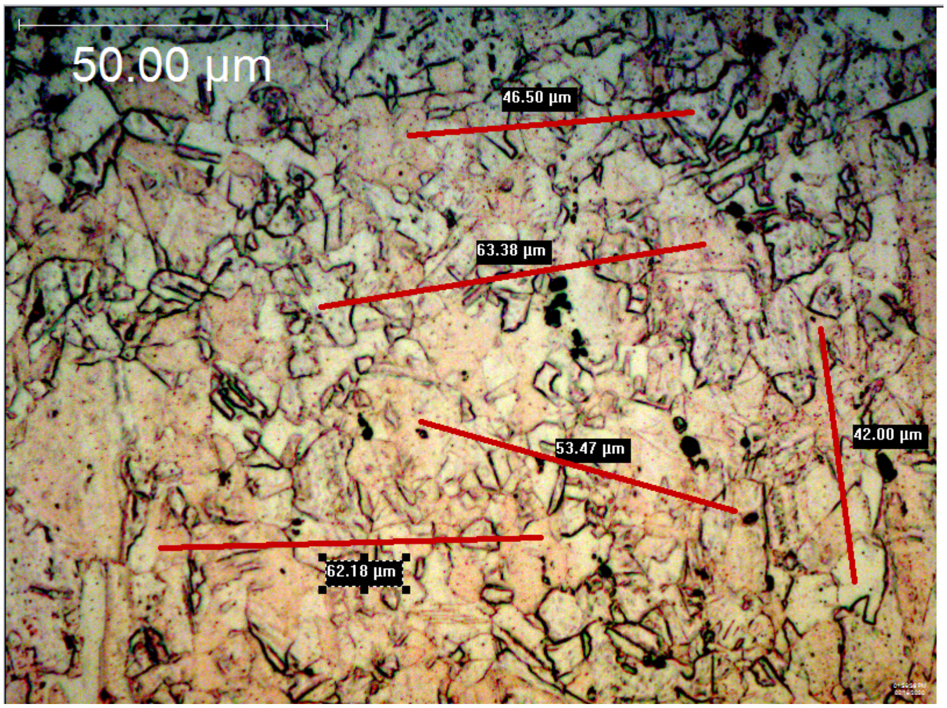

Fig. 3 Copper 18 min annealed sample. This sample required the surface to be etched three times to obtain an image of the quality seen above. Four lines were drawn on the image so the analysis on the average grain size could be calculated.

Figures 4 and 5, show images of 1045 steel under a microscope. Both images are at the same magnification, however, we note that the grain size in Fig. 4 appears much larger. In Table 3, we see that the provided 1045 steel, Fig. 4, has an average grain size larger than the measured grain size for our specimen, Fig. 5. This difference is a result of the heat treatments that our specimen underwent in a previous lab. We annealed the 1045 steel and then rapidly quenched it to room temperature. As a result, lots of small grains were nucleated but did not have time to grow. In addition, the 1045 steel was also tempered which allowed it to recrystallize thus further decreasing the grain size. In Fig. 5, we see a microstructure with many very fine grains. Grain has a strong influence on the strength of a material. As grain size decreased the strength of the material increases as dislocation has a much harder time moving through the microstructure and causing deformation. Thus, we see that Fig. 5 is representative of very strong material. This fact can also be seen numerically in Table 3 as the average grain size in Fig. 5 is significantly smaller than Fig. 4.

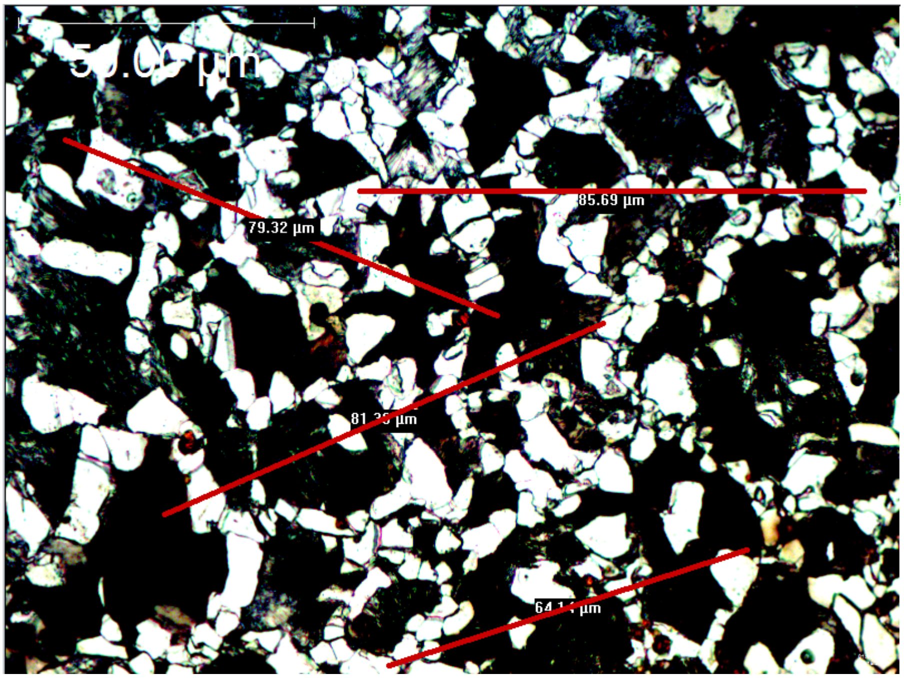

Fig. 4 **1045 Steel sample provided by the lab. This sample was pre-etched so that it could be analyzed while the other samples were being prepared. Four lines were drawn on the image so the analysis on the average grain size could be calculated.**

It can be noted that though 1045 steel underwent the same heat treatment conditions as 1018 steel, we do not see the characteristic needle-like grains of martensite. This is because the steel was tempered after it was annealed. As such, we see a reduction in the amount of martensite present in the microstructure.

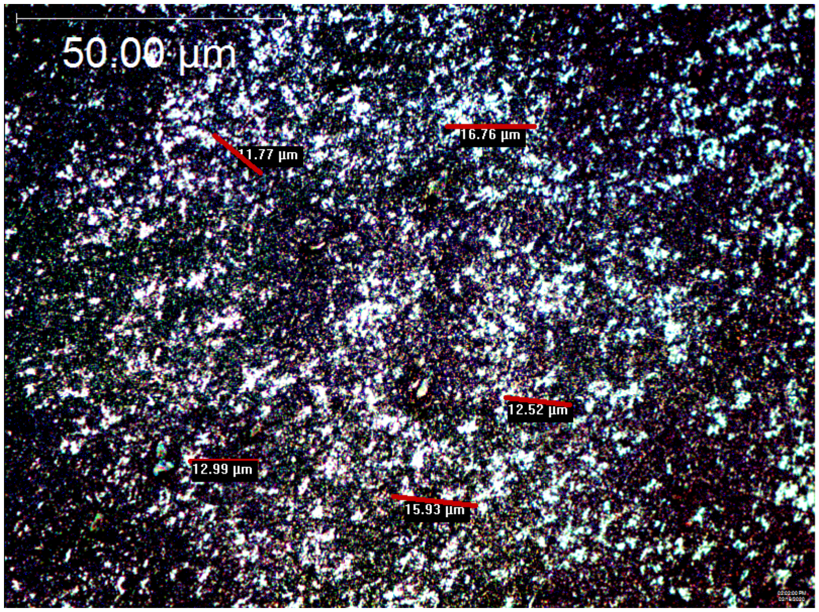

Fig. 5 1045 steel, annealed, quenched, and then tempered prepared in the lab. This image is at the same magnification as the other images (50), thus, it is clear to see that the grains are very small. This is expected for an annealed and rapidly quenched specimen. Five lines were drawn for this image. The addition of the extra line was an attempt to reduce visual counting errors as this sample had the smallest grains.

Figure 6 shows the microstructure of 1005 steel. Similarly to 1018 (Fig. 7), 1005 is low carbon steel, so we would expect similar properties and behaviors to 1018 steel. However, we see initially that 1005 steel has a larger average grain size than the previously examined 1045 steel. Which is to be expected, higher carbon steels are generally harder than low carbon steels so the fact that 1045 has a smaller grain size makes sense. Just as with 1045, we annealed and the rapidly quenched the 1018 steel (Fig. 7). In addition, the 1018 steel was also tempered. The resulting microstructure is a lot of small needles like grains in its microstructure. These needle-like grains and pin dislocations make it very hard for dislocations to move in martensite. This type of microstructure is what we see in Fig. 7: Lots of small needle-like grains that make up the microstructure. This leads to the conclusion that Fig. 7 has transitioned to the martensite phase. The tempering process reduces the concentration of martensite present in a material, which is why we do not see pure martensite.

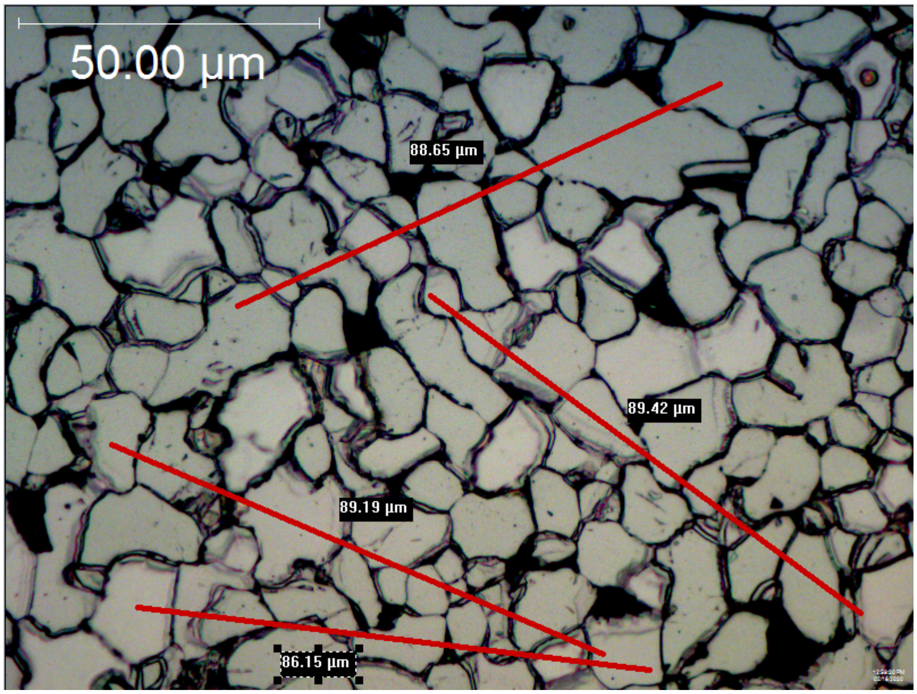

Fig. 6 1005 Steel provided by the lab. This sample was pre-etched so that it could be analyzed while the other samples were being prepared. Four lines were drawn on the image so the analysis on the average grain size could be calculated.

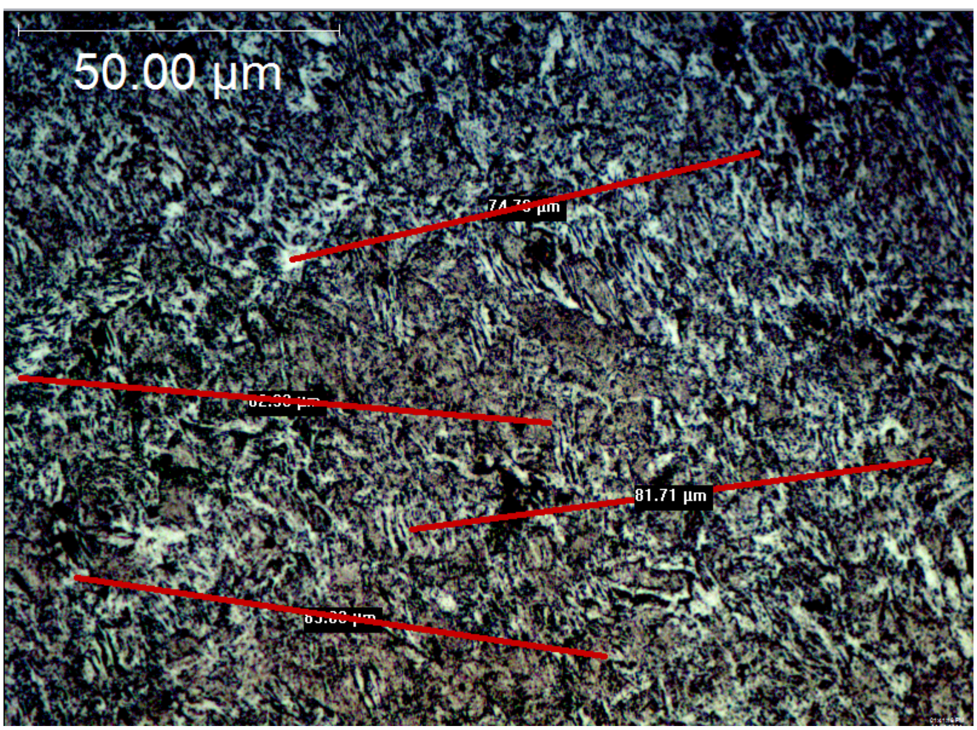

Fig. 7 1018 steel, annealed, quenched, and then tempered prepared in the lab. In this image, the fine needle-like grains that are characteristic of martensite can be clearly seen. This material has a higher concentration of martensite.

Visually it appears that even though the 1045 and 1018 underwent the same heat treatment process the 1018 steel, Fig. 7, has a higher concentration of martensite. However, as can be seen in Table 3, 1045 has a smaller grain size than the 1018 steel.

Conclusion

From the analysis of the microstructures of a variety of materials we observed facts about the composition, properties, and procedures performed on the material. For the copper Fig. 3, we noticed that the average grain size of our specimen was about 52.2 µm smaller than that of pure copper [copper cite]. This difference comes from the fact that our sample had gone through a tempering process for 18 minutes prior to examination. The tempering temperature allows recrystallization to create new grains which decrease the overall average size of the grains.

For the two steels, Fig 5 & 7, we saw that the average grain size is much smaller than the simples provided by the lab. Grain size is an important factor in the strength of the materials. Thus, it was noted that 1045 and 1018 were both stronger than the lab provided samples. This is because of the heat treatment process that both the 1045 and 1018 underwent was the same. Both the 1045 and the 1018 were annealed and then rapidly quenched to room temperature leading to a nucleation dominated process where the result is lots of small fine grains. However, it was also noted that from Fig 7, the 1018 steel appears to have a larger concentration of the martensite phase than the 1045 steel Fig 5.

References

[1] Callister, W. D., and Rethwisch, D. G., 2018, Fundamentals of Materials Science and Engineering: An Integrated Approach, 5th ed. John Wiley & Sons Inc., NJ, Chap 10 & 11.

[2] Spring, K. R. and Davidson, M. W., n.d., "Depth of Field and Depth of Focus," Nikon Microscopy from https://www.microscopyu.com/microscopy-basics/depth-of-field-and-depth-of-focus.

[3] Callister, W. D., and Rethwisch, D. G., 2018, Fundamentals of Materials Science and Engineering: An Integrated Approach, 5th ed. John Wiley & Sons Inc., NJ, Chap 5.

[4] ASTM International, 2013, “Standard Test Methods for Determining Average Grain Size,” E112

[5] Indiamart, 1996, “Olympus, Japan Olympus Industrial Microscope, Model: GX 41”, from https://www.indiamart.com/proddetail/olympus-industrial-microscope-14395887962.html

[6] Song, M., Liu, X, and Lui, L. , 2017, “Size Effect on Mechanical Properties and Textureof Pure Copper Foil by Cold Rolling,” Materials, 10 (5), pp.538

Appendix A

The following table, Table A.1, displays the raw data along with the inspections of the number of intersections for each randomly drawn line on the microscope images for each specimen.

Table A.1 Raw data of microscope photo inspection with counted intersections for each line and calculated average grain boundary lengths for each specimen analyzed.

SPECIMENS	Line Lengths [µm]	Number of Intersections	L_T (Total length of all lines) [µm]	P (Total number of intersections)	M (magnification)	ℓ (Mean grain boundary length) [µm]
110 Copper (Tempered for 18 minutes)	46.50	13	267.53	56	1	4.8
	63.38	11				
	53.47	15				
	62.18	10				
	42.00	7				
1005 Steel (Given)	88.65	9	353.41	42	1	8.4
	89.42	11				
	89.19	13				
	86.15	9				
1045 Steel (Given)	79.32	13	310.50	57	1	5.4
	85.69	21				
	81.35	13				
	64.14	10				
1018 Steel (Tempered Sample)	74.78	45	301.82	177	1	1.7
	62.33	38				
	81.71	47				
	83.00	47				
1045 Steel (Tempered Sample)	11.77	10	56.98	46	1	1.2
	16.76	13				
	12.52	13				
	15.93	10				
	12.99	8				
110 Copper (Tempered for 4 minutes)	98.32	26	375.86	76	1	4.9
	84.24	14				
	98.54	20				
	94.76	16				