Middle East Technical University

Department of Metallurgical and Material Engineering

Mete206 – Materials Processing Laboratory

Experiment 3: Microstructure of Materials

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ABSTRACT

Microstructure are very important in materials engineering as many properties depends on it. In this experiment, effects of the type of material, heat treatment history and mechanical shaping had on the microstructure was observed. It was seen that with increasing C content the amount of pearlite increased. Moreover, it was seen that with the increase in cooling rate the grain boundaries became finer and a crack happened on water cooling of AISI 1060 steel. It was also observed that cold worked material had its grain boundaries align in the direction of the rolling. In the experiment AISI 1060, AISI 1020 and α-brass was used to ready 7 specimens.

INTRODUCTION

A material engineer would like to know the specifics of the material that is being used as to determine whether that material fits into the criteria for his or her design. In the case that failure occurs, an engineer would have to answer the question as to "why" it happened. These and similar situations can be correlated under something which is called as the scope of material engineering.

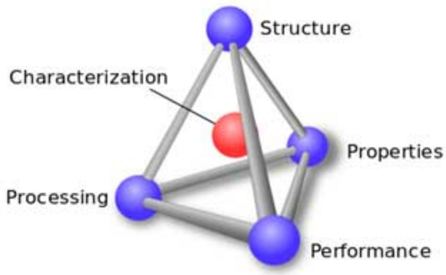

Figure 1. The scope of material engineering (Electrical4U, 2020)

Figure 1 illustrates the scope of materials engineering. What it actually shows is the relationship between structure, properties and processing. Between the three, it can be said that to change one of them another one must also be changed. Microstructure that is one of the three is a very important aspect of the scope. When looked at the above examples that has been made, in the case of failure one of the things that the engineer would first look into is the microstructure of the specimen as the microstructure gives a lot of detail regarding the specimen. As for the Engineer who is doing design, properties are what is very important and microstructure is something that determines what properties a material will have. Therefore, the importance of microstructure can be seen.

There are certain steps involved for the examination of the microstructure. Namely, selecting, sectioning, mounting, grinding, polishing, etching (Laboratary Testing , n.d). In most of the cases there aren't that much space under the microscope to put a specimen under. Therefore, in the case of big metal rods or other large samples where the microstructure is wanted to be known, a representative sample must be obtained by cutting or other methods. This is the step named as selecting and sectioning. In this step while the sample is being obtained by cutting or other methods, there will be damages such as scratches in the surface. There also can be dirt or an oxide layer on top of the surface which is not something that is wanted when looked under the microscope. Therefore there are several steps to make the specimen ready to be looked under the microscope but before that there is the step called mounting where the aim is to protect the materials surface and to fill the voids in porous materials (Akca & Trgo, 2015). Grinding, is the beginning of the three very important steps that readies a material to microscope inspection. It was mentioned that after the cutting operation there will be scratches, dirt, oxide layer and other unwanted things. Grinding allows to clean the surface of the given examples. It is done by the help of multiple sand paper that has different abrasive particles per area. After the grinding is done, the scratches will still exist therefore more procedures must be done on the specimen.

Polishing is what comes after grinding. It has been mentioned that there will still exist small scratches after the grinding process therefore to also eliminate them polishing is done. The difference between polishing and grinding is that the abrasive particles are suspended in a lubricant where else in grinding fixed abrasives are used (Chinn, 2002).

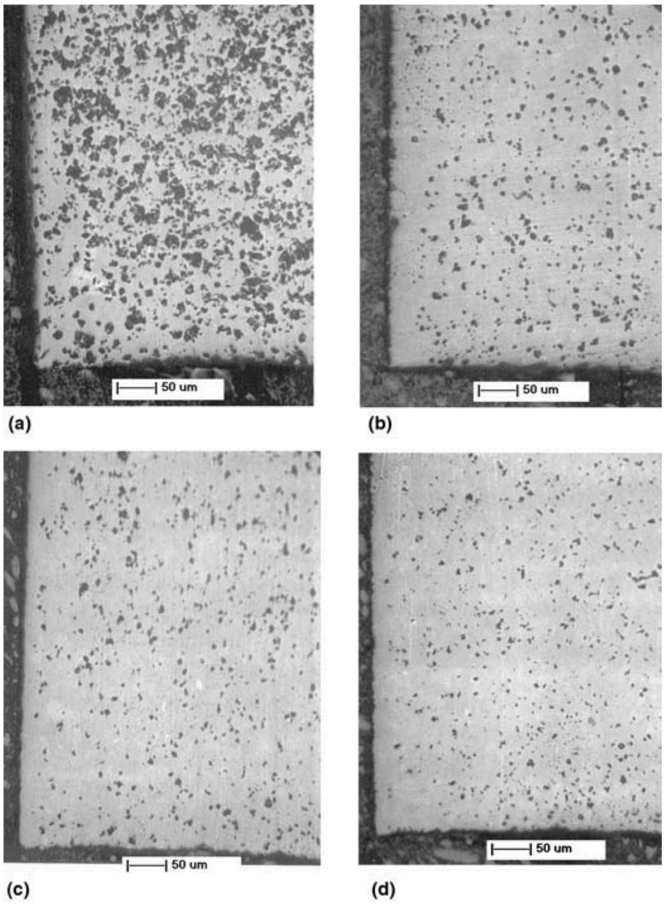

Figure 2. The effects of polishing with increasingly smaller size of diamond paste. (Chinn, 2002)

After polishing the final step of etching is done. When a specimen is highly polished its surface acts as a mirror therefore it can't be looked under the microscope. To be able to see under the microscope etching is done where the specimen is dipped into an acidic solution. One must be careful as to not over or under etch the surface. Moreover, it is important to point out that, grain boundaries will be attacked first in the etching process. After all these steps the specimen is ready to be looked under the microscope.

There are several reasons as to why the microstructures may vary from each other. Firstly, the type of material meaning the composition is a very important parameter in the variation of microstructures. Secondly, the heat treatment also affects the microstructure. Whether the specimen is furnace cooled, air cooled or quenched are determining factors in microstructure. Lastly, mechanical shaping must be talked about. For example, if the specimen is cold rolled the effects of it can be seen by observing the microstructure.

It has been mentioned of the effect of microstructure in determining the materials properties. As engineers, we try to play with the process in order to affect the microstructure therefore the properties. To understand how the microstructure would be like certain diagrams are used. Phase diagrams is one of them and it used in the case of slow cooling. From this diagram if know the composition of the specimen we can understand the phases present at any temperature. The opposite also holds true. TTT diagrams are used when the cooling is not slow. For example, in the case of quenching. TTT stands for time-temperature-transformation. With this diagram once again, the phases can be obtained when the rate of cooling is known.

EXPERIMENTAL

Materials

Table 1. Materials that was used in the experiment

Specimen #	Type of Steel	Type of Cooling
1	AISI 1060	Furnace Cooling
2	AISI 1060	Air cooling
3	AISI 1060	Water cooling
4	AISI 1060	Oil quenching
5	AISI 1060	Furnace Cooling
6	Brass	Cold worked + annealed
7	Steel	Plastically deformed

Equipment

- Optical Microscope
- Etching reagent (2% Nital)
- Grinding Paper
- Lubricant

Procedure

The specimen was cut to allow the sample being viewed under the microscope. As mentioned in the introduction, the steps that were needed to ready the specimen to be viewed under the microscope was done. First, the grinding step. The specimen was grinded with sand paper of different fineness. Namely, 220 → 320 → 400 → 500 → 600 → 800 → 1200. The process was done under flowing water as to not allow it to heat so much as well as to wash away grinded particles so they did not scratch the surface again. Second, polishing procedure. It was done on a rotating disk which had hard abrasive particles. Finally etching was done using the etching reagent 2% Nital. As it can be seen from figure 3, the specimens were readied to look under the microscope.

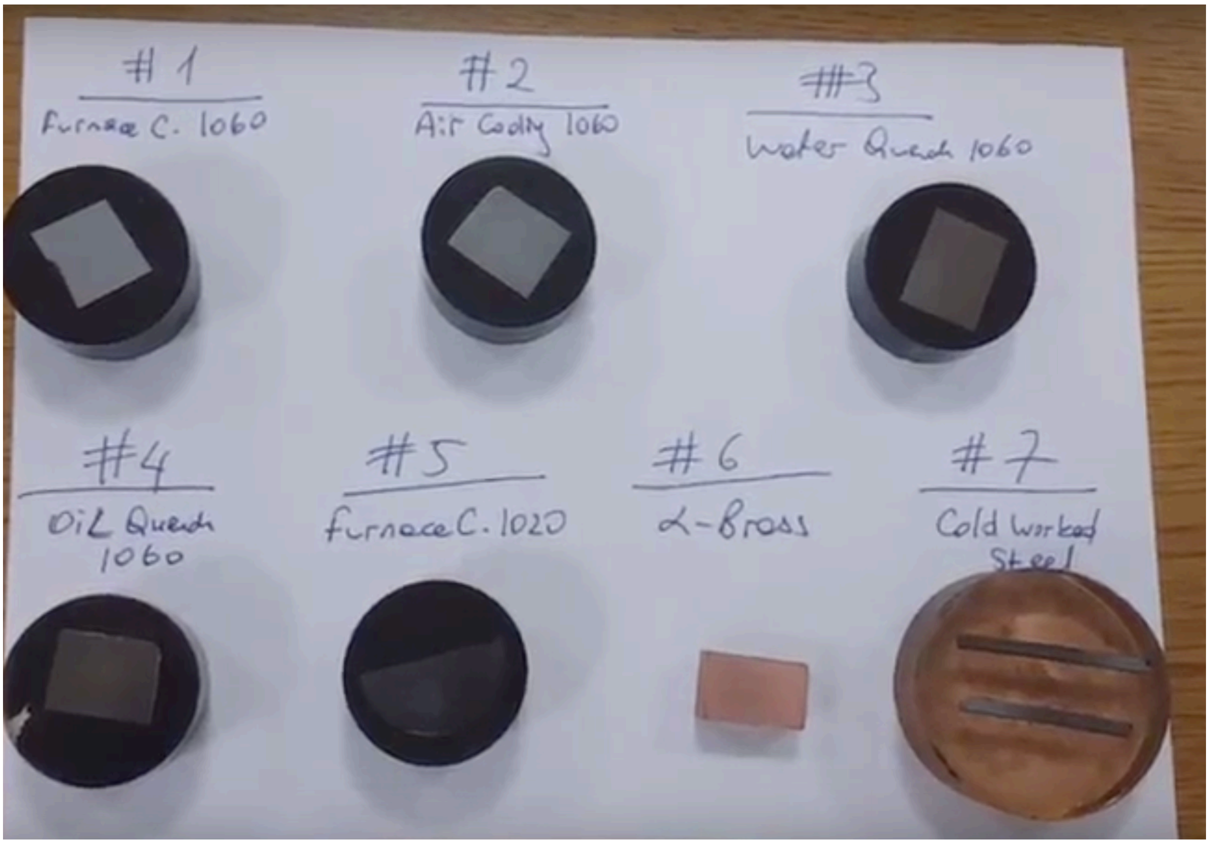

Figure 3. The samples that are looked under the microscope.

To end the experiment all the samples was looked under the microscope.

RESULT

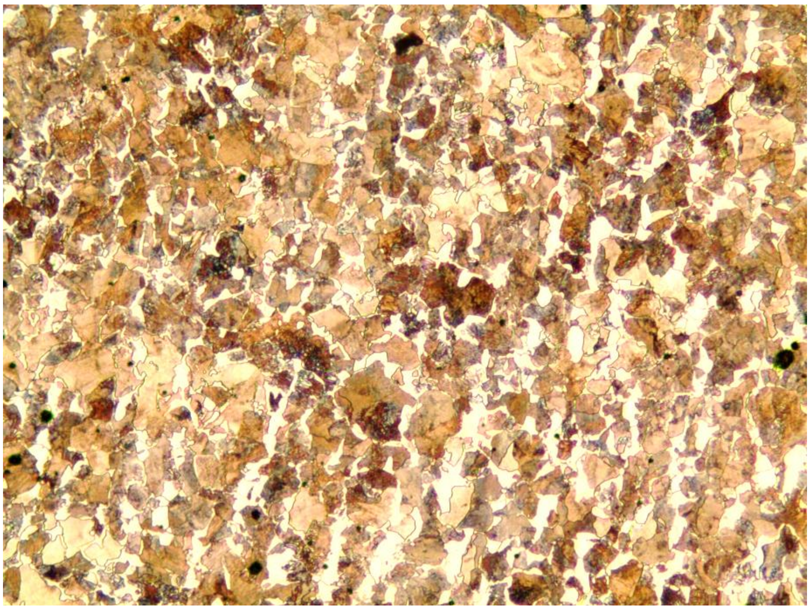

Figure 4. Image taken of sample #1 by the optical microscope at 100x magnification

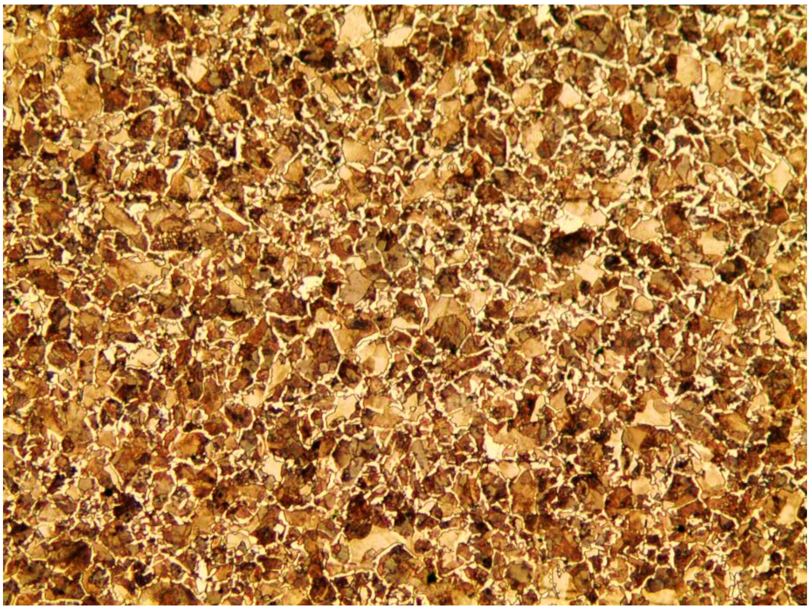

Figure 5. Image taken of sample #2 by the optical microscope at 100x magnification

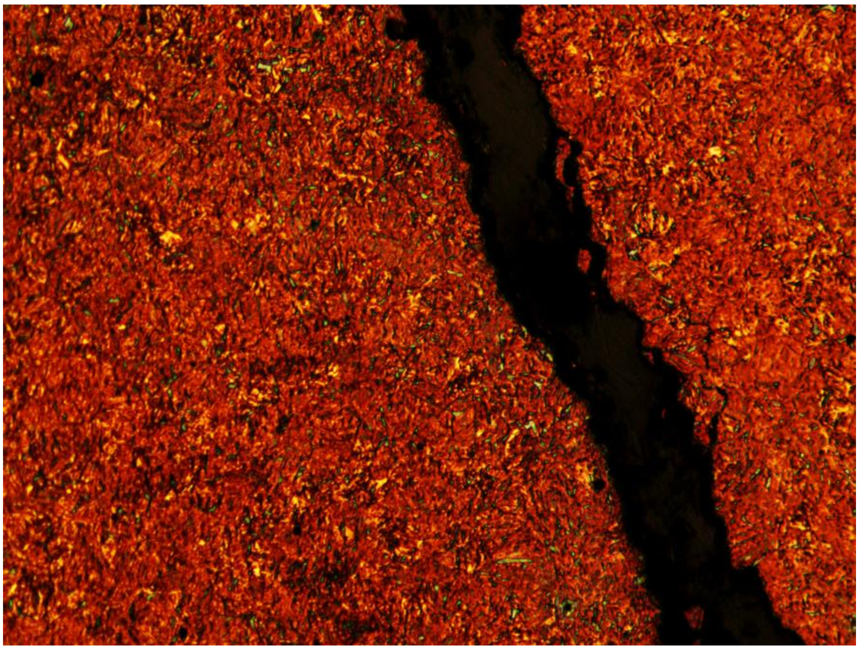

Figure 6. Image taken of sample #3 by the optical microscope at 100x magnification

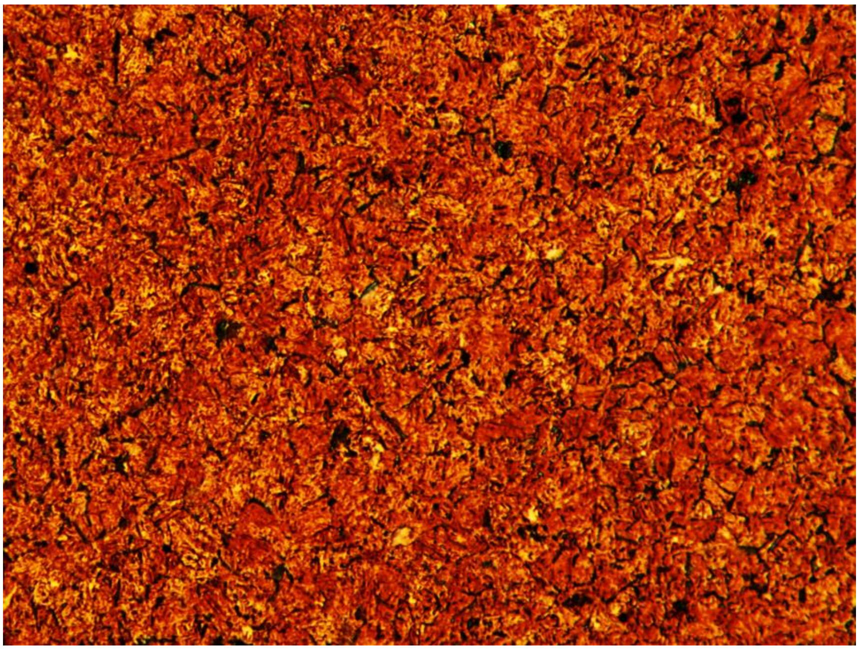

Figure 7. Image taken of sample #4 by the optical microscope at 100x magnification

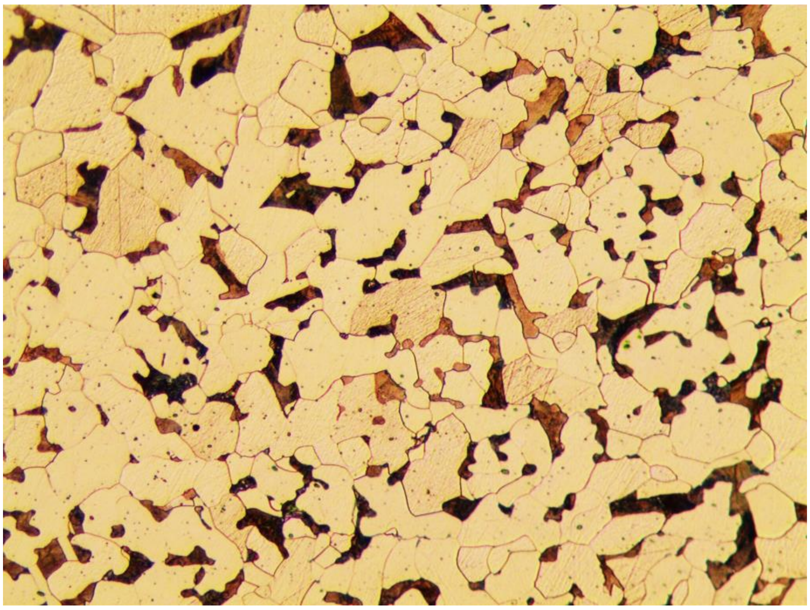

Figure 8. Image taken of sample #5 by the optical microscope at 100x magnification

Figure 9. Image taken of sample #6 by the optical microscope at 100x magnification

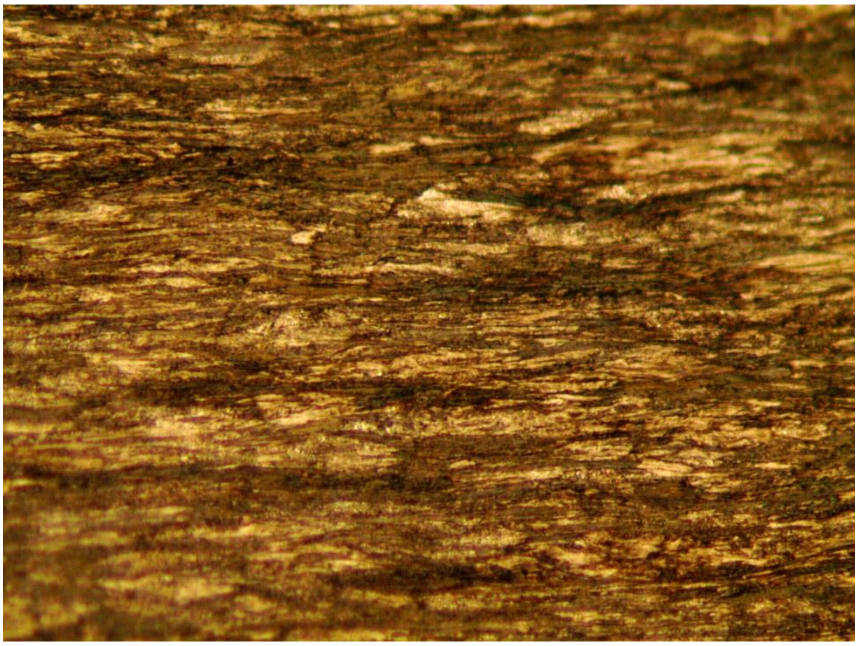

Figure 10. Image taken of sample #7 by the optical microscope at 100x magnification

Figure 4 till 10 gives us the optical microscope images of the specimens from 1 till 7 at a magnification rate of x100.

DISCUSSION

It has been mentioned that etching was done after polishing. The reason is that with only the polishing procedure the surface acts like a mirror meaning that when light is shined on it under the optic microscope nothing can be seen by the user. Therefore, another procedure which is named as etching is done. In the etching procedure, it is known that the grain boundaries will be attacked first. What happens after the attack is that surface grooves appear at the point of grain boundary where when a light is sent there is an image contrast as the light won't bounce back uniformly. This allows one to observe the grain boundaries. It is important to note that if the specimen is dipped into the acid specimen for too long, over etching will occur and if left for too little time than under etching will occur. Therefore, the specimen must be etched just the right amount of time. To give an example to etching of alpha brass which is a single-phase brass, an etchant named as klemm's II reagent can be used where the etchant has a composition of 50 ml saturated Sodium thiosulfate and 5 g of potassium metabisulfite (Pace Technoligies).

In the microstructure of an analysis, a list of steps was given to do. In those steps some are a must and some can be considered optional. The must step are as follows, grinding, polishing and etching. Without these steps it is not possible to examine a specimen under a microscope. The optional steps are sampling and sectioning where if the sample is not big enough to not fit under a microscope then there is no need to further cut it to obtain a sample. Mounting is also not a must as if the handling of the material is already easy there is no need to go through mounting process.

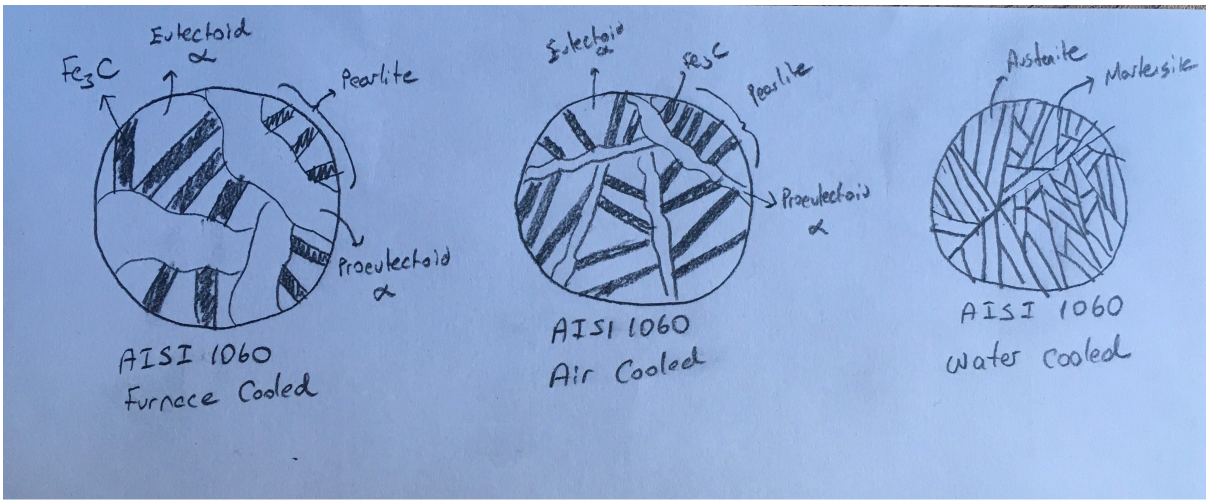

Figure 11. The schematic microstructure of AISI 1060 steel with different rates of cooling

In figure 11, AISI 1060 steel being cooled by furnace, air and water can be seen. Furnace cooling is a very slow type of cooling and it adheres to equilibrium phase diagram. Therefore, for AISI 1060 steel which is a hypoeutectoid steel shows the phases; eutectoid alpha, Cementite and proeutectoid alpha in the microstructure. Moreover, it has course pearlite structure. Air cooling is still relatively slow cooling as the exact phases that is seen a furnace cooled specimen is seen however, there is finer pearlite structure. This makes the air-cooled steel stronger however less ductile when compared with furnace cooled steel. For the microstructure of the water-cooled steel, needle shaped like martensite can be seen as well as austenite that has failed to transform during rapid quench. The water-cooled steel has a structure that is finer than the other two steel which makes it the strongest among them and the least ductile.

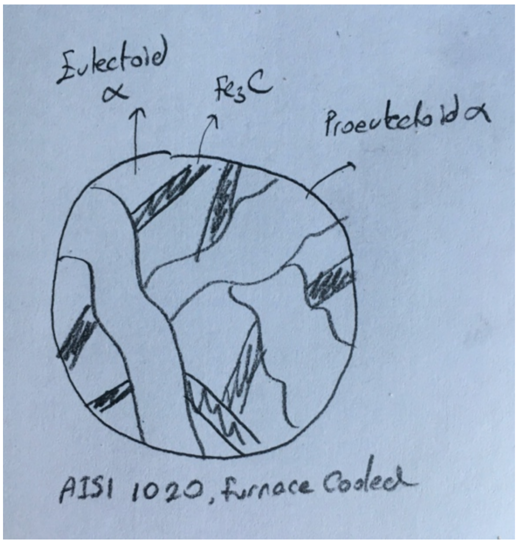

Figure 12. schematic microstructure of AISI 1060 – Furnace cooled steel

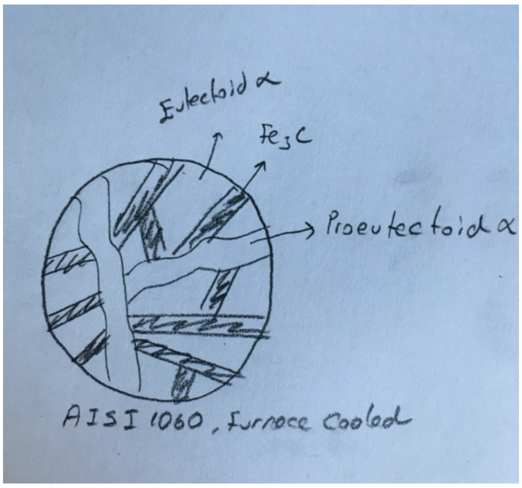

Figure 13. schematic microstructure of AISI 1020 – Furnace cooled steel

In figure 12 and 13 the schematic diagrams of AISI 1020 and AISI 1060 steels that has been furnace cooled can be seen. As they are furnace cooled the equilibrium phase diagram can be used to determine the phases as well as the fraction of each phase using the lever rule. The values that resulted from such calculation were tried to be adhered to during the drawing of figure 12 and 13.

For AISI 1020 steel, it was found that $W_{proeutectoid\ \alpha} = 0.76$ and $W_{pearlite} = 0.24$. Which means that in AISI 1020 steel which was furnace cooled has mostly proeutectoid α in its microstructure. Moreover, $W_{Fe_3C} = 0.03$ and $W_{eutectoid\ \alpha} = 0.76$ being as such means that there is a relatively small amount of cementite inside the structure.

For AISI 1060 steel, it was found that $W_{proeutectoid\ \alpha} = 0.22$ and $W_{pearlite} = 0.78$. Which means that in AISI 1060 steel which was furnace cooled has more pearlite structure than it does proeutectoid α in its microstructure. To add to this, $W_{Fe_3C} = 0.09$ and $W_{eutectoid\ \alpha} = 0.91$ being as such means that there is still a relatively small amount of cementite in the structure but when compared with AISI 1020 steel the amount is increased.

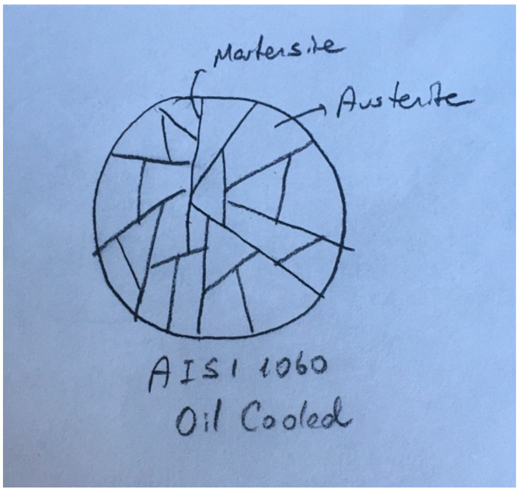

Figure 14. schematic microstructure of AISI 1060 – Oil cooled steel

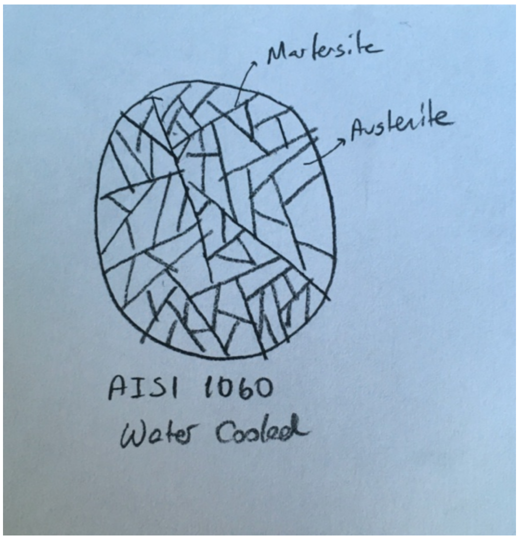

Figure 15. schematic microstructure of AISI 1060 – water cooled steel

Figure 14 and 15 illustrates the microstructure for AISI 1060 steel which has been oil and water cooled. They are both relatively fast cooling mediums therefore needle shaped martensite as well as Austenite which has failed to transform can be seen. However, as water cooling has a faster rate of cooling then oil cooling, its microstructure can be said to be finer than that of oil cooling.

In the optical microscope images the figure 7 correspond to Oil cooled AISI 1060 steel and figure 6 corresponds to water cooled AISI 1060 steel. When it is looked from the microscope there is a huge crack which can be easily seen at figure 6. This crack is also visible when looked with the naked eye to the sample. The reason for this is that austenite's density is denser than martensite so in the case of rapid cooling rates the large pieces cracks by internal stresses (Callister & Rethwisch, 2016). Therefore, it can be said that even with the naked eye one can understand from the crack whether one is water or oil cooled. The problem is most apparent when the carbon has greater than 0.5 wt%. This means that for the AISI 1060 steel it is a problem. To resolve it lower carbon content steel can be used or smaller pieces can be used.

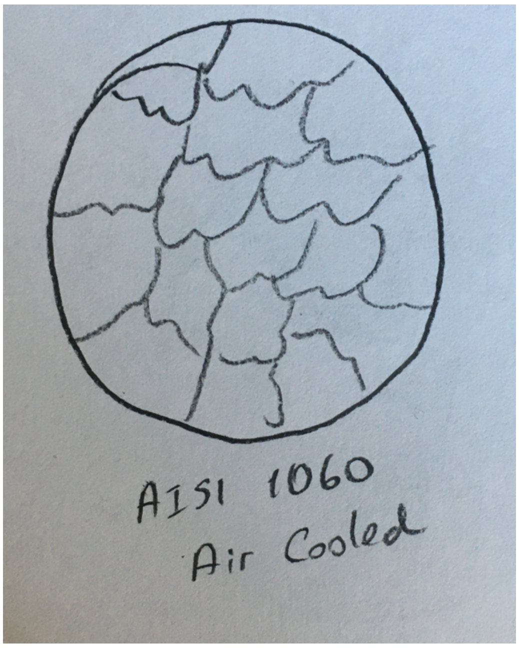

Figure 16. schematic microstructure of AISI 1060 – Air cooled steel

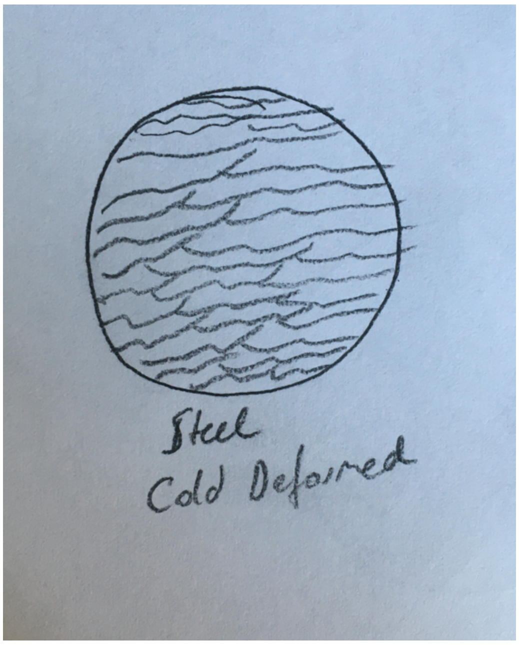

Figure 17. schematic microstructure of AISI 1060 – Cold worked steel

In figure 16 and 17 one can see the specimen #2 and the specimen number #7. Specimen #2 is the AISI 1060 steel which has been air cooled and the specimen #7 is a steel which has gone through the process of cold working.

The difference between them is clearly seen once it is looked at the schematic microstructures that has been drawn in figure 16 and 17. Figure 16 shows us a microstructure of relatively rounded grain boundaries where else in figure 17 where cold work was done, one can see that the grain boundaries have been aligned in the direction of the cold rolling.

In figure 9, the optical microscope image of α- brass can be seen. However, α- brass ha a single phase therefore, one expects it to have only one color but multiple colors are seen in figure 9. The reason can be because the specimen has been cold worked than annealed before looking at it with a microscope. During cold work plastic deformation happens meaning that when it was cold worked it sustained some damages at the surface which can be like grooves. After it was annealed and looked at the microstructure where the light was shined on the specimen there might have been image contrast as the surface is not level. In etching this was the case for the user to be able to see the grain boundaries and similarly these defect make it look like the microstructure has multiple color regions.

CONCLUSION

In this experiment 7 specimen were obtained which had gone through different procedures. It was mentioned that microstructure is very important in determining the properties which engineers had to know to make designs. Therefore, engineers can play with the procedure as to change the microstructure meaning the properties. In this experiment the aim was to observe the effect of cooling rate, c amount, and plastic deformation towards the microstructure. It was observed that when one increased the carbon content the amount of pearlite and the proeutectoid α has changed. Both of them has different effect on the property of the material therefore it is correct to say that this also changed the property of the material. Namely, increasing the C content increased the strength of the material. When looked at the cooling rate it was seen that equilibrium phase diagram was not enough to understand the phases of the material. TTT diagrams were needed. It was also observed that with the increase in the rate of cooling different meta-stable phases occurred in the micro structure and as the rate increased the grain boundaries became much finer making the material stronger but more brittle. For plastic deformation, cold worked specimen was looked at. It was seen that the grain boundaries aligned towards the direction of the rolling. As one can see the process that is done on the material has huge effects on the material itself so it can be said that the materials engineer's job is to find processes that manipulates the microstructure so that desired properties are obtained.

REFERENCES

Electrical4U. (2020, March 14). *Electrical4U*. Retrieved from Electrical4U: https://www.electrical4u.com/electrical-engineering-materials/

Laboratary Testing . (n.d). *Laboratary Testing*. Retrieved from Laboratary Testing: https://www.labtesting.com/performing-microstructural-examination/#.Xo71OS2B1QI

Akca, E., & Trgo, E. (2015). Metallographic Procedures and Analysis – A review. *PERIODICALS OF ENGINEERING AND NATURAL SCIENCES*.

Chinn, R. E. (2002). *Ceramography: Preparation and Analysis of Ceramic Microstructures.* Ohio: ASM International.

Pace Technoligies. (n.d.). *Etchant Database.* Arizona: Pace technoligies.

Callister, W. D., & Rethwisch, D. G. (2016). *Fundamentals of Materials Science and engineering.* Wiley.

APPENDIX

The equations and computations done to find the phase amounts for the AISI 1020 and AISI 1060 steels.

AISI 1020 steel;

$W_{proeutectoid\ \alpha} = \frac{0.76-0.20}{0.76-0.022} = 0.76$ therefore,

$W_{pearlite} = 1 - W_{proeutectoid\ \alpha} = 0.24.$

For the pearlite structure;

$W_{Fe_3C} = \frac{0.20-0.022}{6.7-0.022} = 0.03.$ Which makes,

$W_{eutectoid\ \alpha} = 1 - W_{Fe_3C} = 0.24$

AISI 1060 steel;

$W_{proeutectoid\ \alpha} = \frac{0.76-0.60}{0.76-0.022} = 0.22$ therefore,

$W_{pearlite} = 1 - W_{proeutectoid\ \alpha} = 0.78.$

For the pearlite structure;

$W_{Fe_3C} = \frac{0.60-0.022}{6.7-0.022} = 0.09.$ Which makes,

$W_{eutectoid\ \alpha} = 1 - W_{Fe_3C} = 0.91$