The Effects of Different Quenching Mediums on the Hardness and Microstructures of Steel

by

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ABSTRACT

The rate of heat extraction, hardness, and severity of quenching steel has been investigated using conventional and nonconventional methods. A variety of steel alloys such as 4140, D2, S7, and O1 were used in the preliminary tests with conventional quenching methods to observe the microstructures and hardness development. Results showed that as the cooling rate of the steel increase, the hardness of the steel increased. The microstructure of the conventional quenching methods were similar throughout the metal samples; air quenching produced larger grains of pearlite, ferrite, and cementite, oil quenching produced martensite and pearlite grains, and water quenching produced martensite. Afterwards, a further study was done with unconventional quenching methods on 1045 steel. The unconventional quenching methods that were studied included Olive Oil, Peanut Oil, Quench Fast, and Super Quench, quenchants which have been proliferated through blogs, online forums, and discussion boards. The results have shown that the unconventional quenching produces hardnesses greater than control samples. The quenching methods, however, within the range of 50 Vickers Hardness of each other, which is a small difference. Super Quench and water quench samples are within the standard deviation of their samples set from the hardness of each other similarly to Olive Oil and Quench Fast. Additionally the two groups of similar hardness had similar microstructures being composed of mainly martensite. It could be concluded that water and Olive Oil have the same effects as Super Quench and Quench Fast on 1045 steel respectively; therefore water and olive oil can be used as substitution providing a solution that are sustainable, nontoxic and quick to prepare.

The metallurgical community has proliferated the use of unconventional quenching media as methods to yield superior results in steels. However, the results in this work show that the usage of different media rather than conventional quenching media, produce hardness and microstructures that are similar in value and composition. Therefore, there is no advantage to using quenching methods that have been advertised through blogs, online forums, and discussion boards.

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1. Introduction:

The microstructure of steel is one of the major influencing factors that contributes to its hardness, brittleness, toughness and other characteristics. These microstructures can be predicted and obtained by manipulating the rate that the steel is cooled at through quenching, which is the rapid reduction of temperature of a material by submerging the material into a media with a lower temperature. Quenching steels is the first step in the heat treatment of steels to obtain the desired characteristics and microstructures. In a typical heat treatment process, quenching is usually followed by annealing. Annealing involves heating the material above its recrystallization temperature, maintaining a temperature for a suitable amount of time, and then cooling which decreases thermal stress and allows atoms to migrate in the crystal lattice decreasing the number of dislocations. Annealing can affect the strength, ductility, and hardness.[1]

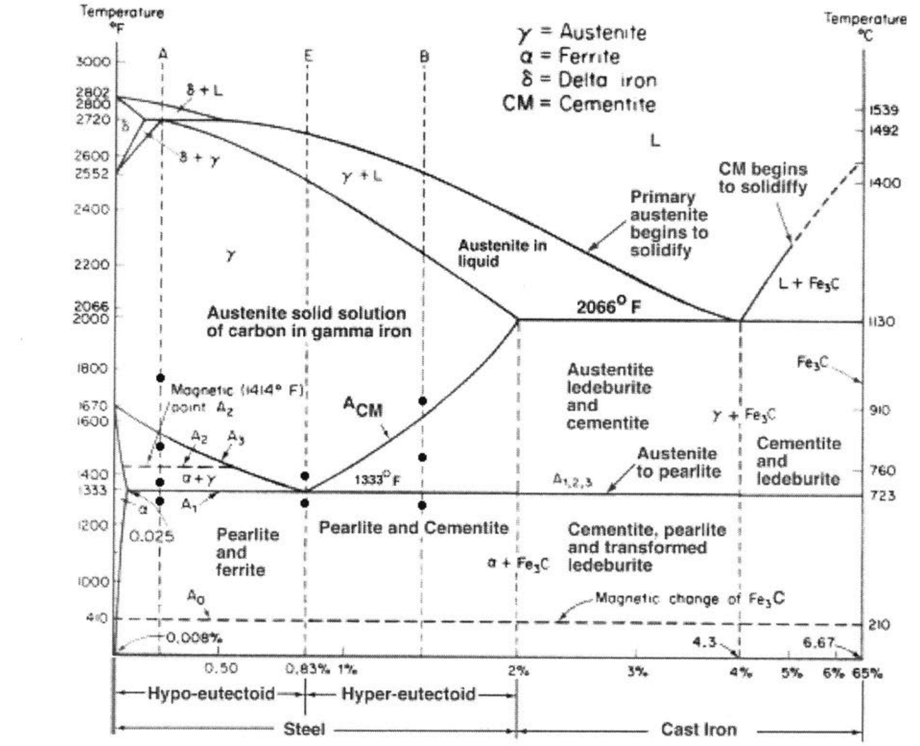

Figure 1-1: This chart shows how the carbon content in steel is able to affect the microstructure composition of steel when the steel is cooled slowly.

The quenching process works by heating steel to a high temperature, where the microstructure transforms to the phase austenite. The steel is then removed from the heat and submerged into a fluid to cool the steel. The reduction in temperature can transform the austenite into different phases such as pearlite, cementite, ferrite, bainite, retained austenite, and martensite. The phases that the austenite is transformed into are dependent on the cooling rate and contribute to different material characteristics[2].

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[1] Verhoeven, John D., *Steel Metallurgy for the Non-Metallurgist,* (Ohio: ASM International, 2008.) pg 116

[2] (Verhoeven 2008, 9)

Many of the techniques for the heat treatment of steel were originally developed in the late 19th and early 20th century through empirical tests. Because of this empirical collection of data and practices along with the modern proliferation of these procedures through blogs, online forums and discussion boards there has been a spread of misinformation about the techniques, and quenching mediums that can be utilized to obtain optimal results. Therefore, this study was designed to quantify these claims and procedures that have proliferated within the metallurgical community, examining the results of quenching in air, water, and oil for W1, 4140, 01, and A2 steels. Furthermore, the steels will have their cooling rates measured within the cooling media and then compared to the hardness and structure suggested with their corresponding TTT (time-temperature-transformation) diagrams. A second study with 1045 steel will be conducted to understand and quantify the effects of unconventional quenching media compared to conventional media by quenching the steel with water, soybean, peanut, olive oil, and mediums known as Super Quench and Quench Fast. The resulting pieces will be subjected to hardness tests, viewed underneath a microscope to understand the influence of each medium on its microstructure, compared to their TTT diagrams suggested structure, and analyzed to understand the validity of any the claims of the metallurgical practices of non-conventional quenching mediums.

1.1 Background:

1.1.1 Steel and Cast Irons

Steel is an alloy of iron and carbon in its simplest form. Most modern steels contain over 95 percent iron with the remaining percentage being carbon or other alloying elements. Iron that has carbon levels below 2.1% per weight are considered steel and irons with percentages above 2.1% wt. are considered cast iron [3]; these experiments will concentrate on steels that have carbon content lower than 2.1%.

Steel can be thermally manipulated to create variation in the microstructures (or phases) such as pearlite, ferrite, cementite, bainite, martensite, and austenite. The percentage of carbon that is dissolved within the iron also affects the microstructure of the steel, and the ability for certain phases to form[4]. These microstructural changes in steel contributes to the mechanical behavior of the material such as hardness, toughness, ductility, and strength.

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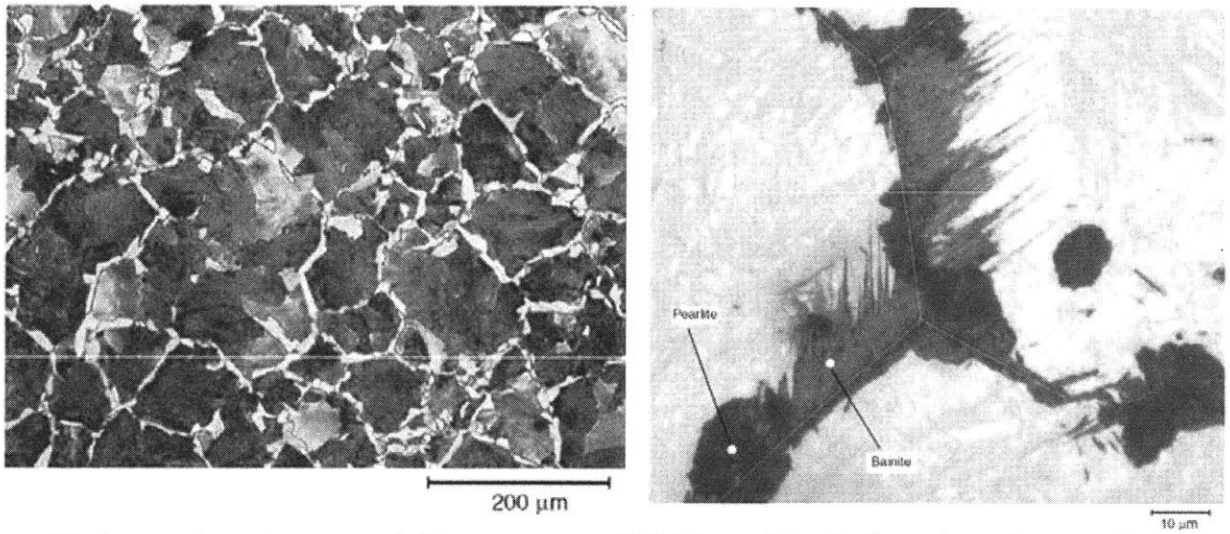
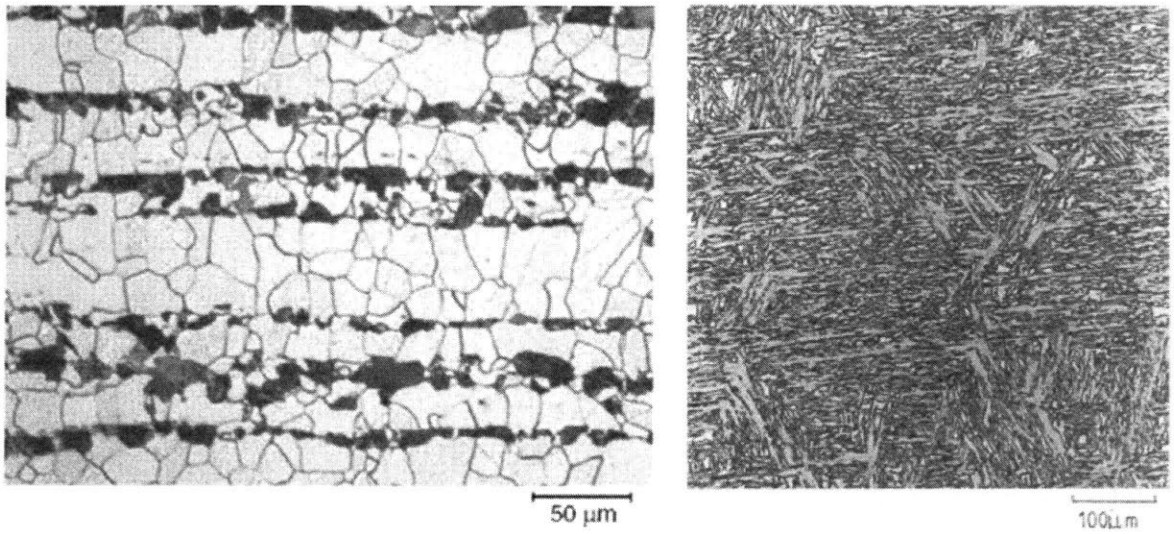
[3] (Verhoeven 2008, 9)

[4] (Verhoeven 2008, 10-11)



a) Dark austenite grains surrounded by white pearlite grains with a magnification of 100X achieved by Nital etching

b) Bainite and Pearlite formed on prior austenite grain boundaries indicated by white lines in 1095 steel at 1000X magnification



c) Microstructure of steel consisting of Ferrite as the White grains, and pearlite as he dark grains at 240X magnification

d) Martensite observed in the microstructure of low-alloy steel at 150X Magnification

Figure 1-2: Displays the different phases that can be present in alloy steel. At magnifications from 100X to 1000X. The images display, austenite grains, pearlite, bainite, ferrite,[5] and martensite[6].

1.1.2 Lattice Structure

The individual carbon atoms are incorporated into the iron crystal lattice by occupying interstitial sites between the iron atoms of the austenite structure (face centered cubic) or ferrite (body centered cubic)

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[5] (Verhoeven 2008, 24,25,32)

[6] (University of Cambridge n.d.)

at high or low temperatures respectively. Atoms smaller than 42% of the iron atom diameter generally occupy interstitial sites but because carbon atoms are about 56% of the iron diameter they push the iron atoms apart a small amount.[7] As more carbon is dissolved the further apart it pushes the iron atoms, therefore there is a limit to how much carbon can be dissolved into the iron. The solubility limit for carbon in austenite can be seen as a line labeled A_{CM} in Figure 1-1, the line gives a maximum amount of carbon that can be dissolved in austenite at a specific temperature. Alloys that have carbon percentages greater than the line presented are in a two-phase region that will consist of a mixture of austenite and cementite grains[8].

Crystal structure is a factor that plays into how much carbon can be dissolved in steel. Steel has three primary crystal structures which are Face Centered Cubic (FCC), Body Centered Cubic (BCC), and Body Centered Tetragonal (BCT).

The lattice and crystal structure of the steel affects the amount of carbon that can be dissolved. At low temperatures, below A1 and A2 on the phase diagram, has a body centered cubic (BCC) crystal structure, BCC iron has a lattice parameter (the length of the side of the cube) of 0.285 nanometers[9]. At high temperatures above A1 and A2 ferrite transforms into austenite, which has face centered cubic (FCC) crystal structure, with a lattice parameter of 0.355 nanometers to 0.360 nanometers[10]. The larger lattice of FCC allows carbon atoms to occupy octahedral interstitial sites in the crystal structure. Octahedral interstitial sites of BCC ferrite are smaller for carbon than that of FCC austenite due to face centered cubic structures having more distance between their atoms than body centered cubic[11]. This is why solubility of carbon in ferrite is much smaller than solubility of carbon in austenite. When additional carbon is added to austenite, the solid solution has the same FCC crystal structure as in pure iron with the only change being the distance between the iron atoms.

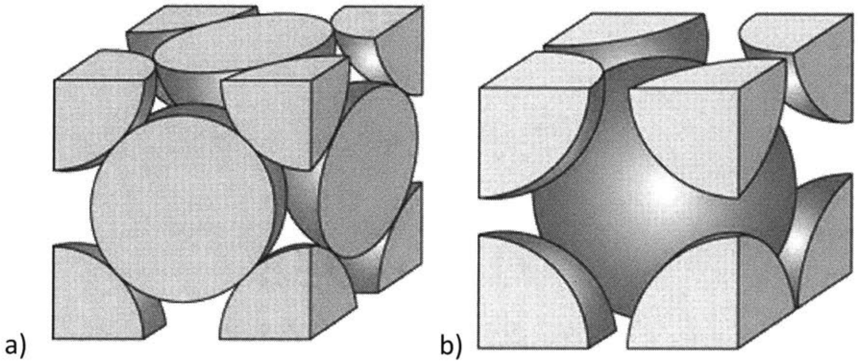

Figure 1-3: This figure demonstrates the cubic structure that can be expected from a) face centered cubic strucutures and b) body centered cubic structures

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[7] (Verhoeven 2008, 10-11)

[8] (Verhoeven 2008, 15)

[9] (Onink, et al. 1993)

[10] (Onink, et al. 1993)

[11] (Verhoeven 2008, 10-11)

1.1.3 Steel Phases

When the steel is heated to a temperature above the austenization temperature (usually between 900 and 1100 Celsius) the grains transform into austenite grains, similar to a phase change in water[12]. This phase change is an endothermic process, therefore the temperature of the steel will stay close to transformation temperature until all the grains are transformed.[13] Austenite, however, is never seen in plain carbon steel at room temperatures, the only exception being when high percentage carbon steels are quenched rapidly producing mixtures of martensite and retained austenite.[14]

Conversely, certain phases of steel will only start to form from the austenite if cooled extremely slowly. The iron carbon phase diagram in Figure 1-1 displays the variety of phases that can be formed in steel with different carbon percentages in its composition. When the steel is subjected to slow cooling processes, the composition of steel is able to be predicted and controlled by solutions and phase diagrams transforming the austenite to desired or expected ferrite, pearlite, and cementite combinations. The excess carbon soluble in austenite, but with limited solubility in ferrite, becomes incorporated into phases of cementite, or pearlite, which is a layered structure of ferrite and cementite which can be seen in Figure 1-4 that has a large magnification factor allowing for a view of the different layers. The entire area underneath the austenite temperature is a two phase region if cooled slowly being composed of ferrite and cementite.[15]

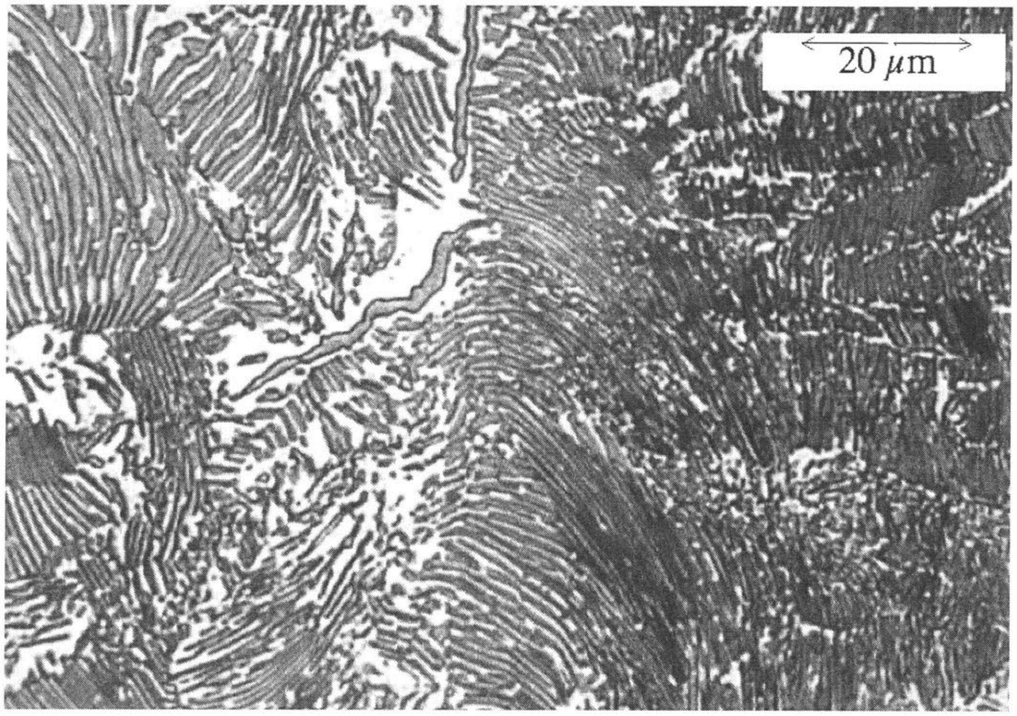

Figure 1-4: The figure shows the pearlite microstructure in steels at a magnification factor of 500. The cementite and ferrite appear to be a stack of layers.[16]

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[12] (Verhoeven 2008, 7)
[13] (Verhoeven 2008, 6)
[14] (Verhoeven 2008, 15)
[15] (Verhoeven 2008, 14)
[16] (University of Cambridge n.d.)

Steels can be classified into three different categories, hypoeutectoid steels where carbon levels are below the eutectoid point (0.83% Carbon, point E in Figure 1-1), eutectoid steels where carbon levels are at the eutectoid point, and hypereutectoid steels where carbon levels are high past the eutectoid point.[17]

Within the different categories the microstructure of steel can vary widely in their combination of pearlite, ferrite, and cementite due to their percentages of carbon content. Each of the phases contain a different carbon content, ferrite essentially being pure iron because of its composition being 99.98 percent or purer with respect to carbon, cementite existing in only one composition, Fe_3C, with a composition of 6.7 wt. % carbon, and pearlite having a composition of 0.77% C.[18]

Hypoeutectoid steels will produce a combination of pearlite and ferrite in the microstructure. Since ferrite can dissolve almost no carbon, when austenite transforms and produces ferrite virtually all the carbon atoms in that volume element must be ejected into any remaining austenite increasing its carbon content. The austenite composition will have been raised to the eutectoid composition of 0.77% carbon creating pearlite when cooled.[19]

Eutectoid steel, if cooled slowly, will be composed of primarily pearlite having a composition of 0.77% C. Pearlite is a microstructure that is composed of layers of ferrite and cementite whose plate thickness depends on the cooling rate. In air cooled samples the cementite plates of pearlite are so thin they often cannot be seen in an optical microscope, usually having thickness less than 0.2 micrometers. Ferrite plates are much fatter than the cementite plates, occupying 90 percent of the volume compared to the 10 percent for the cementite. At the pearlite grain boundaries, there is an abrupt change in the orientation. Unlike cementite itself, pearlite is not brittle due to the fine size of the cementite plates.

In a hypereutectoid steel, the microstructure will consist of a combination of cementite and pearlite. For example, 1095 steel, which has 0.95% Carbon, upon cooling from austenite will start forming thin plate shaped grains of cementite on the prior austenite grain boundaries at 760 Celsius, and the austenite grains will have a composition of approximately 0.85%. The original composition of 1095 austenite grains are reduced to 0.85% C after the cementite forms because the cementite, at 6.7% C, must absorb surrounding carbon atoms as it forms.[20] Cooling further to A_1 temperature of 727 Celsius more cementite is formed and the austenite composition drops further to the eutectoid composition of 0.77% C. Cooling the steel below 727 Celsius transforms the austenite grains to the pearlite structure that will not change on further cooling. As the amount of cementite increases due to the increasing carbon content, the brittleness and hardness of the material will also increases.

In each case the set of prior austenite grain boundaries are filled with a proeutectoid phase. The phase diagram in Figure 1-1 provides information about the volume fraction. For example 1075 steel has a 0.75 percent carbon composition which must be nearly all pearlite and small part ferrite. Microstructures with rates of air cooling or slower will have a structure mixture of ferrite and cementite, where the cementite is almost entirely present in the form of pearlite. A pearlitic steel is desired for applications that requires a balance of strength and toughness.

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[17] (Verhoeven 2008, 23-24)
[18] (Verhoeven 2008, 24)
[19] (Verhoeven 2008, 24)
[20] (Verhoeven 2008, 27)

When steel is cooled rapidly from the austenite region its phases, amounts, or compositions can no longer be estimated from the phase diagram, as it is not allowed the time to reach equilibrium conditions. The rapid cooling prevents the austenite from transforming into a ferrite and cementite structure and instead martensite is formed. If the transformation is forced to occur very rapidly by quenching there is not enough time for the carbon atoms to diffuse, so some or all get trapped in the ferrite causing its composition to rise above its 0.02 percent solubility limit. This causes a distortion from its bcc form which is called martensite. Martensite has a body centered tetragonal (bct) crystal structure which increases its carbon content and its hardness. The strength and hardness of the martensite is found to increase dramatically as the carbon percentage of the steel increases. Verhoeven compares the chemical bonds holding the iron atoms together as springs, developing on the analogy stating that as the carbon content increases the springs will are extended by larger amounts thereby making it more difficult to extend them further and therefore making the structure harder.[21]

The new phases that develop in the microstructure upon cooling occur at the boundary lines of the past phase. Meaning that martensite, ferrite, pearlite and cementite begin form where two grains of austenite meet. The location of formation for these phases influence the characteristics of the steel.

1.2 Characterization Techniques

1.2.1 Optical Microscopy

Optical microscopes work by using reflected light to generate an image. With a smooth surface, a larger fraction of the incoming light is reflected back up the image path and therefore creates a bright white image.[22] Etching, which removes material from the polished surface at different rates, allows for different amounts of light to be reflected back up the image path depending on the phases present. The rate of removal depends on both the crystal orientation of the grain and the type of grain. Grains that are single phase, such as ferrite, austenite, and cementite, will have their atoms removed uniformly from point to point so an originally polished surface will remain smooth within a given grain after etching.[23] Therefore single phase grains appear at the white end of the gray-level range and it is not often possible to distinguish between them by appearance without further information. Pearlite, however, will generally appear gray to black in an optical microscope with air cooled pearlite appearing solely dark. Pearlite appearance in a microscope is due to the layers of cementite being etched slower than the layers of ferrite; Cementite being very fine layers scatter the incoming light away from the image path and generate a darker image. If the spacing of the cementite plates are large enough, the cementite plates will appear as dark lines with white ferrite plates between them.[24] Martensite's most common variation is lath martensite which when viewed under a microscope appears as fuzzy.

1.2.2 Hardness Testing

Hardness testing is a non-destructive test of mechanical properties; having different units of measurement such as Rockwell, Brinell, and Vickers methods. Vickers hardness were used in this study, which is made with a diamond indenter in the form of a pyramid. The diamond indenter is pressed into the metal with a fixed load, the hardness is then calculated utilizing the average diagonal length and the

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[21] (Verhoeven 2008, 27)

[22] (Verhoeven 2008, 21)

[23] (Verhoeven 2008, 21)

[24] (Verhoeven 2008, 22)

load used with a formula that then calculates the Vickers hardness number. However when taking hardness measurements, the material that is being tested must be taken into account. Depending on the material that is being tested its indent will vary such as pearlite would indent more due to its lower hardness than cementite.[25]

1.2.3 CTT and TTT Diagrams:

To predict the percent of phases within certain steels that will occur through different cooling rates Time temperature transformation (TTT) and continuous cooling transformation (CCT) diagrams are often utilized.

CTT diagrams apply to steels cooled at slower rates such as occurs near the center of steel samples without thin cross sections. By measuring cooling rate and finding the corresponding cooling rate on the diagram, ferrite, pearlite, or bainite can be estimated. The diagrams are able to show when 10, 50, 90, and 99% of the austenite will be gone. There is an alternate form of this diagram that describes progress only at the center of the bar. With these diagrams it is possible to determine the microstructures that will be present only at the center of round bars for quenches of water oil and air.[26]

To avoid pearlite formation and obtain 100% martensite in this steel the temperature must fall below a certain temp in a certain time frame.

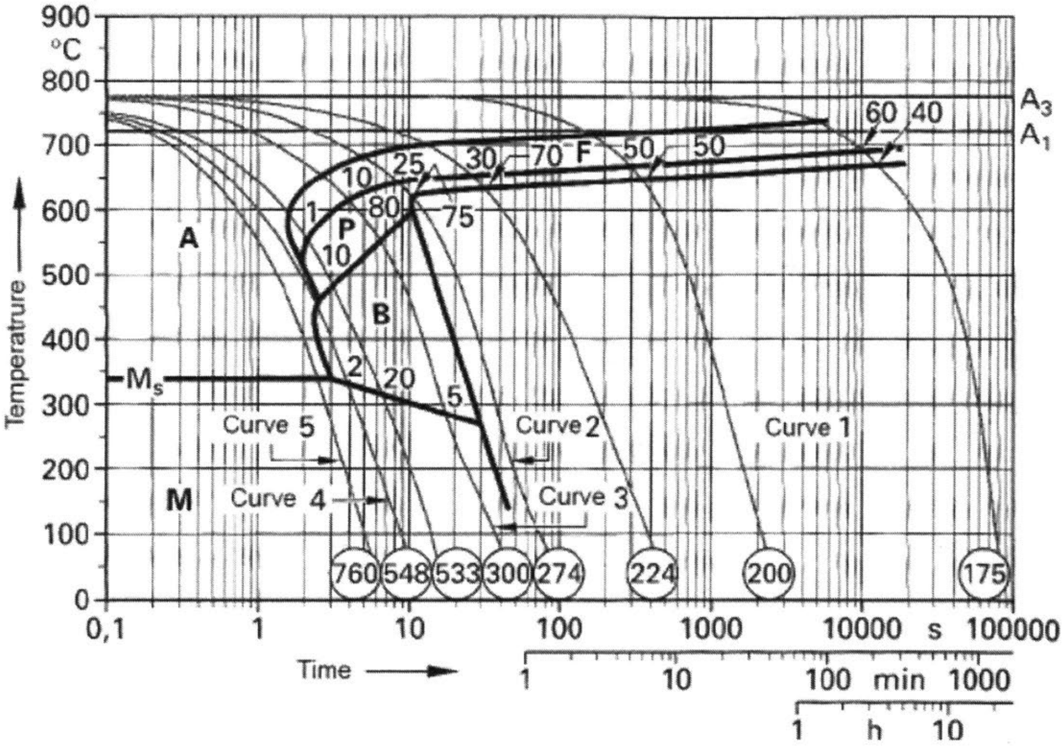

Figure 1-5: The TTT diagram shows the rates when different phases of steel are produced in 1045 steel relative to its temperature and time elapsed after quenching has

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[25] (Verhoeven 2008, 24-27)

[26] (Verhoeven 2008, 50)

started in a logarithmic time scale. There are bold letters in the diagram which are abbreviations of phase such as P-Pearlite, B- Bainite, A-Austenite-M-Martensite

1.3 Quenchant Background

1.3.1 Unconventional Oil Quenchants Background:

To verify or debunk the substitution of conventional quench media with unconventional media in the development of hardness of steel, it is important to understand previous experiments. The quenching media that will be compared and analyzed for substitution will be Super Quench for water and soybean oil, peanut oil, and olive oil, for Quench Fast (also known as 11 second oil) which is mineral oil based.

Mineral based oils have found to show cooling capacities comparable to vegetable based oils. Mineral based oils have been argued to be relatively expensive, toxic, and non-biodegradable and thus generate negative environmental effects[27]. Using vegetable fatty based oils as a replacement has been researched to offer a sustainable and biodegradable substitution. The results have shown positive effects on the mechanical and microstructure properties of steels as oils being used as quenchants. Oil quenchants were able to produce moderate grain structure with moderate homogeneity between pearlite and martensite. Lower viscosity oils were able to produce higher heat transfer coefficients and therefore the heat flux during quenching was influenced by viscosity. Studies have shown that excellent hardening properties have been obtained with locally available oils.

1.3.2 Super Quench Background:

Super Quench's effectiveness was developed and tested in the National Metal and Material Technology Center. A mixed quenchant consisting of brine, typically made by adding sodium chloride or calcium chloride to distilled water, and surfactants was applied in the quench hardening process on AISI 1015. The different variations of quenchants included water, brine (7% Salt level), heavy brine (15% Salt level), and heavy brine with dish washer liquid. The results of the experiment showed an almost doubled cooling rate than water with salt levels of 10% or higher, with or without a surfactant. The hardness of AISI 1015 clearly improved in brine solutions with Heavy brine and dish washer liquid producing increased surface and core hardness.[28] The results lead to the conclusion that quenching in heavy brine and dish washer liquid, also known as Super Quench, improve core hardness while simultaneously improving surface hardness compared to quenching in water.[29]

Additionally, Super Quench is a solution that has a reputation in online forums for being a quenchant that contributes to even cooling and high-speed quenching. The solution is composed of five gallons of water, five pounds of salt, 32 ounces of Dawn dishwashing liquid, specifically blue and if concentrated 28 ounces is requested, and eight ounces of Shaklee Basic or seven ounces of unscented Jet-Dry. The science that is behind Super Quench's success is unsupported and by hearsay using phrases such as "The Jet-Dry... does something chemically to the surface of the steel. It allows the salt in the mix to start attacking as it hits the air.... These surfactants are wetting agents. They break down the surface tension of water allowing it to make contact with a material."[30] There is a lack of academic papers testing the

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[27] (Shinde and Rao 2013)

[28] (KORAD, et al. 2011)

[29] (KORAD, et al. 2011)

[30] (Brown n.d.)

validity of Super Quench's claimed properties. Other properties that Super Quench has been advertised to help with include the prevention of a Leidenfrost layer with the metal due to the influence of wetting agents[31]. Another incorrect assumption that forums have stated has been that the increase of salt in the solution increases the specific heat of the water but in actuality salt decreases the specific heat of water creating a slower heat exchange between quenchant and steel.[32]

1.4 Influencing Factors:

1.4.1 Carbon Levels

The amount of carbon that is dissolved into the iron influences the material properties. If the steel has carbon content levels that are greater than the eutectoid point, it is likely that the material will have a higher hardness because it has higher levels of cementite, a phase which is harder than ferrite although more brittle.

There are important effects that occur as the percent carbon is increased in plain carbon steels such as the CCT and TTT curves shift to the right, meaning that the hardenability of plain carbon steel increases as the percent carbon increases. Additionally, as the percent carbon increases the M_s temperature, the temperature where martensite begins to form, decreases as well. With Table 1 in mind, we can theorize that D2 steel will have the highest hardenability, but will need the largest heat flux to obtain it, and conversely 4140 and 1045 will have the lowest hardenability but will need the lowest heat flux. However, not all steels that are being experimented with are plain carbon steels and the alloying elements that are in the composition of the steels may influence the hardness and hardenability of the material.

Carbon Content and Equivalent Carbon Content in the Composition of Varying Steels

	4140	1045	S7	O1	D2
Carbon Content	0.36-0.46%	0.43-0.50%	0.45-0.55%	0.85-1.05%	1.40-1.65%
Equivalent Carbon Content	0.64-0.93%	0.53-0.65%	1.35-1.88%	1.10-1.48%	3.9-4.83%

Table 1-1: This table shows the carbon content in different steels and the variation that the steel can have. Additionally it shows the equivalent carbon content that is due to additional elements in the composition of the steel.

1.4.2 Cooling Rate:

Variation in cooling rates produce different microstructures within the steel and therefore produce different material properties when they are in room temperatures. With steels that are slowly cooled from the austenite area, the combination of phases can be estimated from the cooling graph and consequently their strength and properties can also be estimated. However, if the steel pieces are cooled at a fast enough rate they avoid the formation of ferrite, cementite, or pearlite and instead the austenite transforms into martensite which contributes to a harder steel than the slow cooled process.[33]

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[31] (Brown n.d.)

[32] (Brown n.d.)

[33] (Verhoeven 2008, 89)

The cooling rate that steel experiences largely depends on the media that is being used for quenching. Specific heat, a critical influencer for the rate at which the heat transfers between the materials, varies between media. Since the same steel will be used for the different experiments, and its specific heat stays constant in the heat transfer, the specific heat of the media dictates the rate which the heat will be transferred. A media that has a greater specific heat will be able to conduct the heat transfer at a faster rate compared to a media with a lower specific heat. Table 2, then suggests that Super Quench and Water should have the highest heat transfer with small variations between each other while air should have lowest heat transfer rate.[34] Additionally, from the table its observable that Peanut and Olive Oil have specific heats that are very similar, meaning that their cooling rates and possibly end products will be very similar.

Specific Heat of Media (kJ/(kg K)								
	Air	Water	Peanut Oil	Olive Oil	Soybean Oil	Transmission Oil	Quench Fast	Super Quench
Specific Heat	0.718	4.186	2.03	1.97	1.97	2.13	1.67	4.008

Table 1-2: This table shows the specific heat of different media that are used for the steel quenching. The higher the specific heat of the media, the higher the heat flux between steel piece and quenching media takes place. Quench Fast specific heat has been approximated to be to that of refined mineral oil as McMaster does not provide its specific heat and Transmission Oil has been approximated to petroleum as Automatic Transmission Oil is petroleum based. [35]

During the process of heat transfer there can be phase changes in the media that is being used to quench the steel. If there is enough energy dissipated during the heat transfer it will allow for media to experience the phase change from liquid to gas at the surface of the steel. The phase change that happens to media at the surface of the steel creates a drop in the rate of heat transfer, which is due to gases lower specific heat than liquids. The phenomenon that happens between the media and steel is called a vapor blanket or the Leidenfrost effect. [36]

To minimize the effects of this vapor blanket, agitation needs to be added to the piece when it is quenched.[37] The agitation of the piece promotes the movement of liquid in the bath, minimizing the phase change as different liquid obtains the heat. The purpose for agitation is to also distribute the heat in the media so that the heat transfer can be at its greatest at all time during the quenching process.

The rate at which temperature drops is fastest in the surface and slowest at the center therefore it is possible to have the outer layer be pure martensite while the center will have pearlite and bainite making the hardness of the bar lower in the central regions.

1.4.3 Grain Size:

To improve hardenability and consequently the hardness of the steel it is necessary to force the start of the phase formation curves to greater times. Increasing the formation time, means that it is more difficult for ferrite, pearlite and bainite to form in the austenite because there is larger amount of time

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[34] (Engineering ToolBox 2003)

[35] (Engineering ToolBox 2003)

[36] (Verhoeven 2008, 125,129)

[37] (Verhoeven 2008, 127)

that the steel can be cooled before producing those phases. The start formation curves can be affected by increasing grain size. [38]

New austenite grains will nucleate on the boundaries between cementite and ferrite with their subsequent growth rate controlled by carbon diffusion in the freshly formed austenite. First formed austenite grains will have small diameters and grow rapidly as the temperature increases, therefore small grains can be effectively retained by using the rapid cyclic heat treating. [39]

Product constituents virtually always form on the austenite grain boundaries. A larger grain size will reduce the amount of grain boundary per unit volume which in turn will improve hardenability. Grain size will not influence the cooling rate, however it will affect the position of the P_s and P_f, pearlite start and pearlite final respectively, curves. The presence of larger grains will cause a shift to in the formation curves creating softer material properties. [40]

Fine grained parts are needed for improved toughness and the increased hardenability of the alloy steels allows much deeper hardening than is possible with fine grained plain carbon steels.

2 Experimental Design:

2.1 Heating and Quenching

Prior to starting any heat treatment for the different steels, control samples were taken of each steel to understand the initial properties. The 0.25" by 0.5" cross section steel pieces will be heated in a coal forge until they reach 1200 degrees Celsius. When the steel reaches this high temperature it transforms to austenite. The steel pieces will then be taken away from the forge and be submerged into different media that will quench the metal. When quenched, the austenite will transform into ferrite, pearlite, cementite or martensite depending on its rate of cooling. The rate which the metal cools at is dependent on characteristics of the media such as the specific heat, quantity, initial temperature, and Leidenfrost temperature. Therefore, the media which will be used for the quenching process will be prepared on the side at a room temperature to imitate the conditions that the medium would be presumed to be used in, with a minimum quantity of 5 gallons of the media available for heat distribution and to maintain the heat equilibrium close to the starting point. When the steel is submerged into the media, it will be agitated to prevent the influence of the Leidenfrost effect. The formation of a vapor blanket between the piece and the media influences the rate of cooling and therefore the transformation of the phases in the steel due to convections slower heat transfer than conduction. The cooling rate of the steel will be measured for each different media used and will be compared to the TTT diagrams of the metals.

We will test O1, D2, 4140, and S7 steels with conventional mediums such as water, transmission oil, and air. These tests will allow us to understand the fundamental effects that conventional media will have on the microstructure of the steels along with its hardness. We will conduct a further study with 1045 steel using the mediums water, air, olive oil, peanut oil, vegetable (soybean) oil, and a media known as *Super Quench* and *Quench Fast 11 second oil* as quenchants. *Super Quench* is a solution comprised of 28 oz. of Dawn blue dish washing detergent, 5lb of salt, 5 gallons of water, and 8 oz. of JetDry that is said to be an

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[38] (Verhoeven 2008, 45,53)

[39] (Verhoeven 2008, 110)

[40] (Verhoeven 2008, 89)

incredible fast quenchant. The heat treatment will consist of heating up the metal to a temperature of 1200 Celsius, submerging the hot end of the metal into the quenching media, agitating the piece to prevent the formation of a vapor blanket, keeping the piece submerged until it reaches room temperature.

2.2 Analyzation

After the steel pieces have been subjected to the heat treatment they will be cut into 3 different plane orientations consisting of XY, YZ, ZX which can be seen in Figure 2-1. These pieces are then mounted into bakelite, ground to a grit of 4000, and polished to 0.5 micron alumina. The mounting and grinding polish is to produce surfaces that are flat and even which can be later etched and tested for hardness without inconsistencies in grain heights.

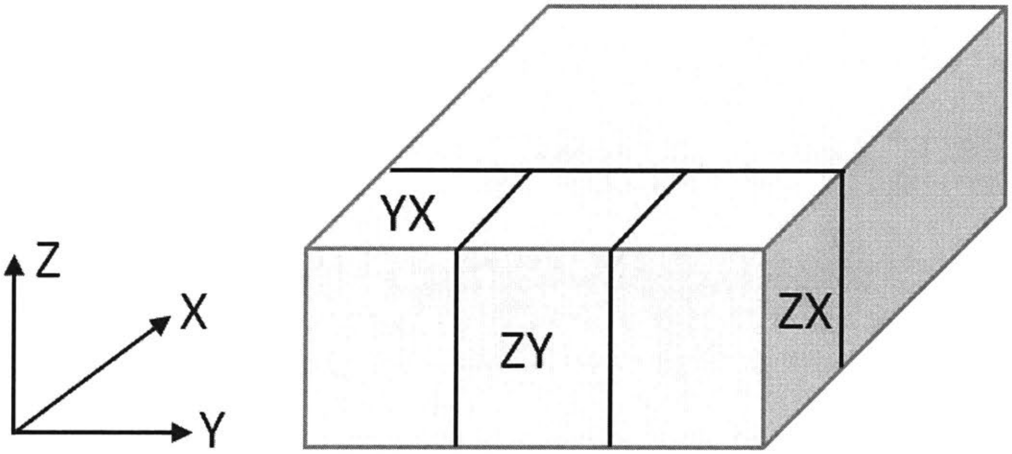

Figure 2-1: The diagram shows the different plane orientations samples that the steel will be cut into to analyze hardness and microstructure.

The pieces are cut into the different planes to view the microstructures of the metal from different angles. Viewing these different planes allow us to see how the rolling process affects the grain sizes of the metal. The steel samples will be tested for their hardness using the Vickers hardness scale procedure. A diamond tip will be pressed into the metal and the diagonals of the indentation will be measured to calculate the material's hardness. There will be three indentations done on each of the materials that will be four indentation widths away from each other to eliminate influence from past indentations. The indentations will happen at the edge, the center and the in between the edge and the center of the steel pieces to understand how the media affected the hardness throughout the steel piece and to understand if there were any temperature gradients when cooling. After hardness testing the pieces, they will be subjected to etching by a nital solution that will dissolve a layer of metal from the surface and allow us to look into the microstructure of the material. The nital solution allows us to view the phase composition of the microstructure, due to different phases that compose the steel reacting to the nital solution at different rates and therefore reflecting different amounts of light when observed under a microscope.

3 Results and discussion:

3.1 Conventional Quenching Methods

The control experiments that were conducted reacted to the heat treatment and quenching as expected, developing hardness that correlated with the cooling rate with the exception of D2 steel. As the cooling rate increased due to the different quenching media, the hardness of the steel increased, with the amount of hardness increase depending on the type of steel; these results, which can be seen in Table 3, correlates with discussions on the relation of cooling rates and hardness.[41] There was one exception to this relationship which happened in D2 steel with the oil quenched steel being softer than the air quenched sample. D2's air quenched sample was expected to have a greater hardness than oil due to being an air quenching steel however, the increase in hardness for its water quenched sample was unexpected. In figure 3-1, the different hardness that were produced due to the different quenching methods were graphed by increasing specific heat; the hardness can be clearly seen increasing throughout the different types of steel in the same manner as the specific heat of the media increases.

O1	Trial 1	Trial 2	Trial 3	Average	St Dev
As Received	185	181	180	182.0	2.6
Air Quenched	418	421	408	415.7	6.8
Oil Quenched	698	728	723	716.3	16.1
Water Quenched	750	734	731	738.3	10.2

D2	Trial 1	Trial 2	Trial 3	Average	St Dev
As Received	190	180	187	185.7	5.1
Air Quenched	734	746	757	745.7	11.5
Oil Quenched	591	599	605	598.3	7.0
Water Quenched	803	811	811	808.3	4.6

S7	Trial 1	Trial 2	Trial 3	Average	St Dev
As Received	137	173	180	163.3	23.1
Air Quenched	662	698	695	685.0	20.0
Oil Quenched	754	750	738	747.3	8.3
Water Quenched	842	824	847	837.7	12.1

4140	Trial 1	Trial 2	Trial 3	Average	St Dev
As Received	230	250	246	242.0	10.6
Air Quenched	295	304	306	301.7	5.9
Oil Quenched	478	470	511	486.3	21.7
Water Quenched	649	634	655	646.0	10.8

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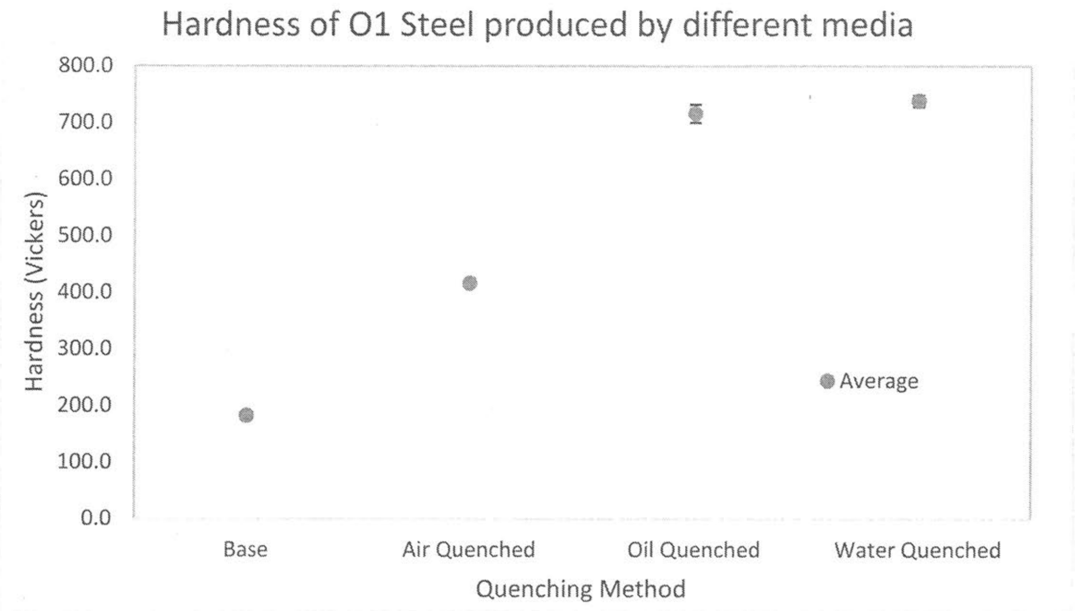
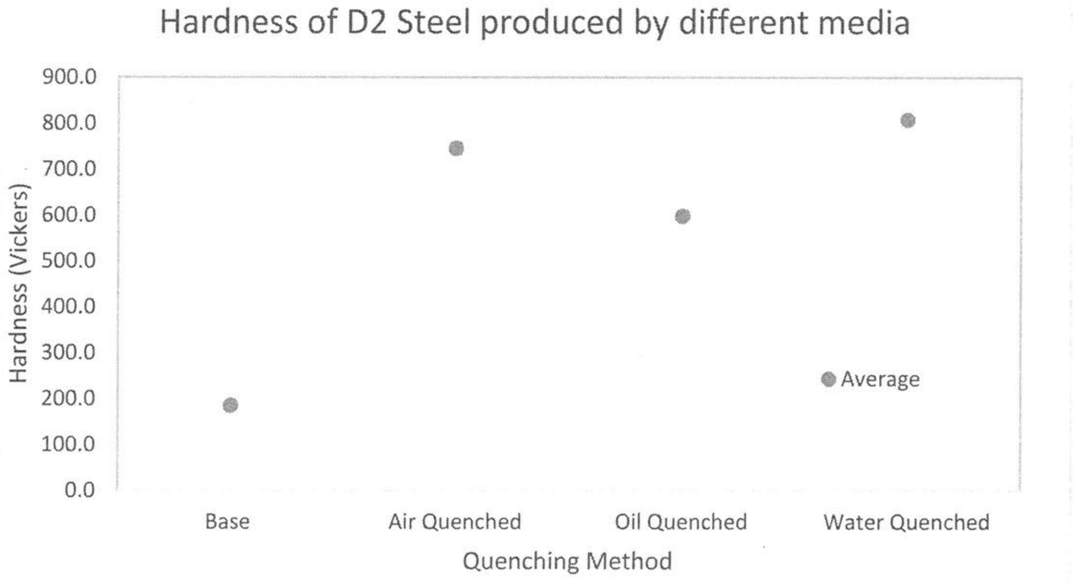
[41] (Verhoeven 2008, 15)

Table 3-1: The tables shows the Vickers Hardness of steel samples after they have been heat treated and quenched in different media in three different locations of the sample. The varying steel hardness have been averaged and their standard deviation calculated for analyzation purposes.





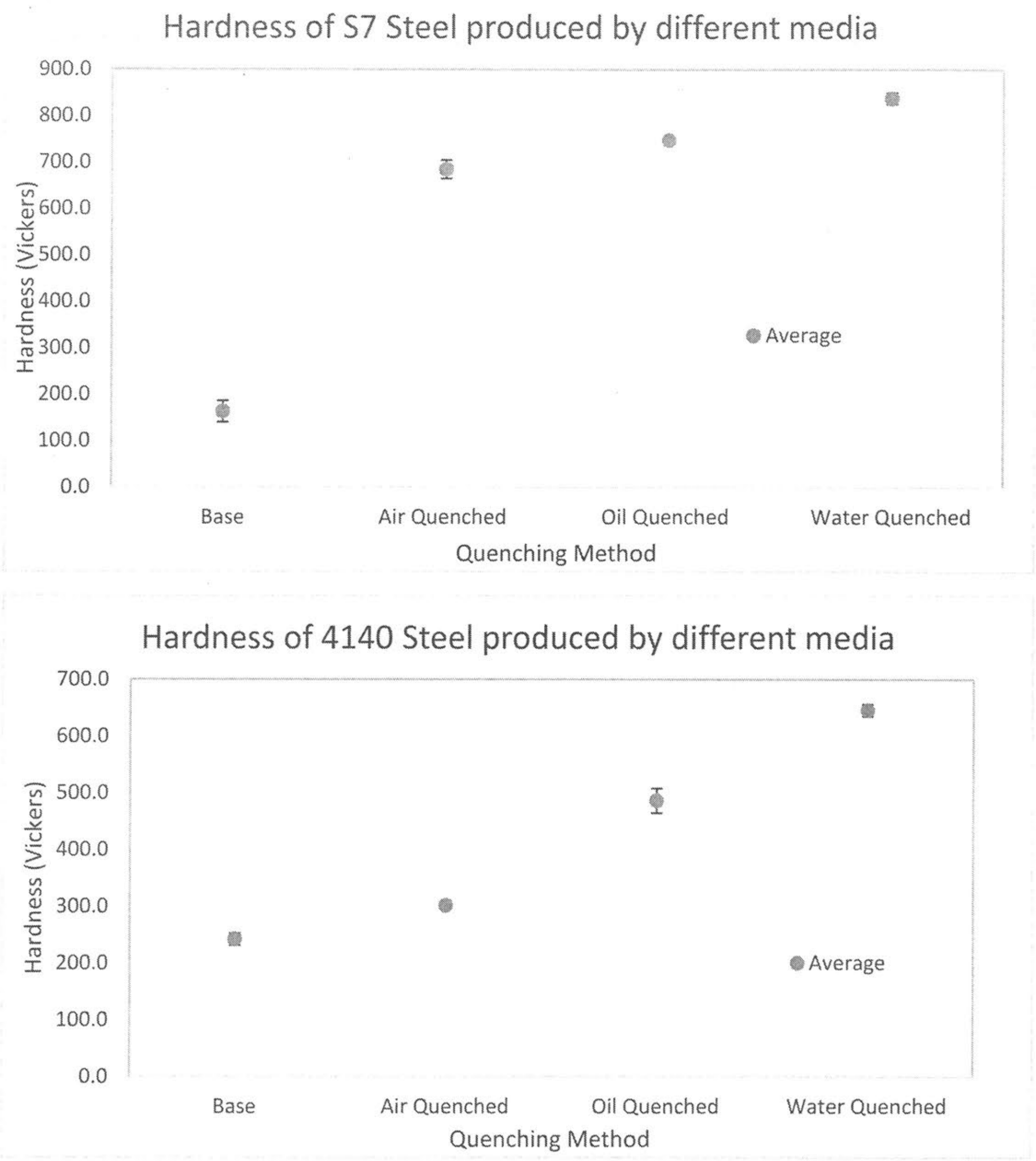
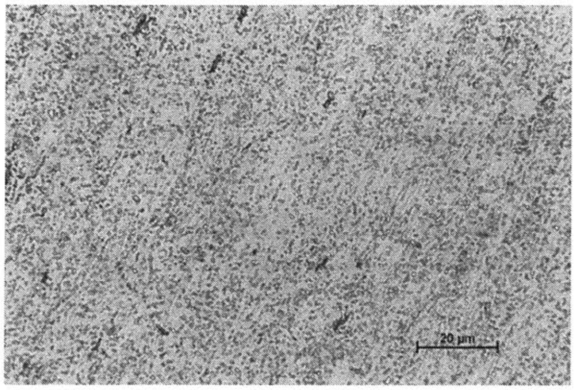
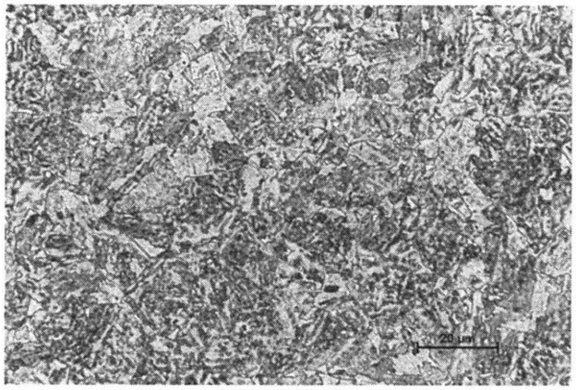


Figure 3-1: This graph demonstrates the hardness of conventional quenching on 4 different types of steel that vary in carbon content and base treatment. The standard prediction is that steel will become harder as the media increases its cooling rate, which can be seen in all samples except for D2 where the air quench is higher than the oil quench method.

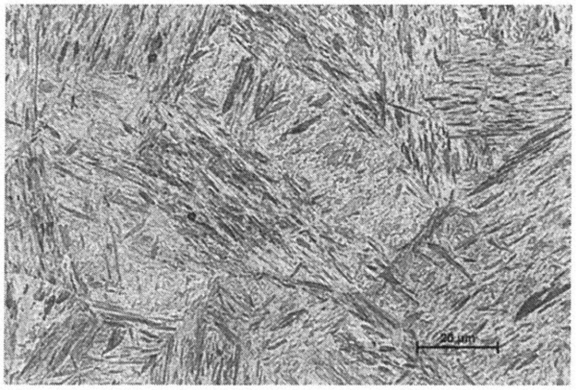
The microstructures of the steel samples were observed under a microscope to understand the effect of the different quenching media on the steel's composition and see the variation that the same quenching process had on varying steels. With Figure 3-2 we are able to analyze the control steel quench samples together and see the similarities in the observed microstructures. In the four different types of steel we are able to see that spheroidized microstructures are present most likely to aide in the machining process of the steels[42]. In a,c, and d of Figure 3-2 we are also able to observe that water quenching was able to produce microstructures that were composed of martensite which would contribute to a high hardness that was also observed in hardness testing. In the water quenched microstructure of b in Figure 3-2, there appears to be areas that are large grains of white that are probably come from fresh martensite and are surrounded by martensite this might be due to D2 having the highest carbon content of the tested steels. Furthermore, the oil quenched steel samples from a, c, and d in Figure 3-2 have a microstructure that seems to be composed of martensite, and larger amount of fresh martensite or grains that produce a white appearance in the microscope like ferrite or cementite. The observed air quenched microstructures in Figure 3-2 shows the steel samples similarity for having larger distinguishable grains that are comprised of cementite, pearlite, and ferrite.



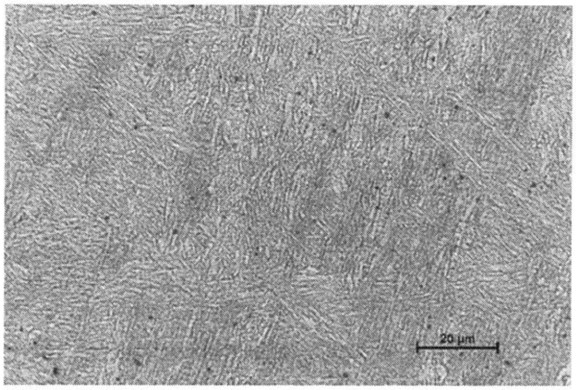
a) 4140 Control



4140 Air Quenched



4140 Oil Quenched



4140 Water Quenched

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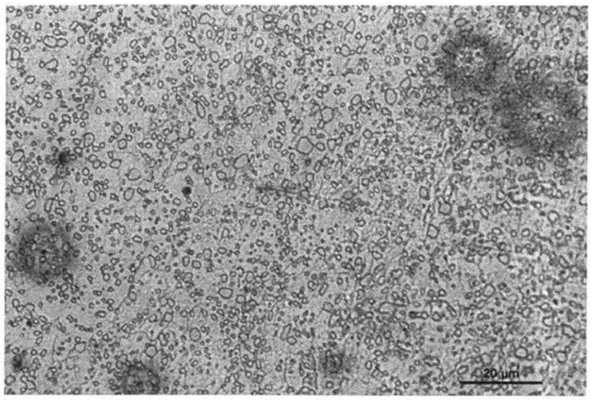
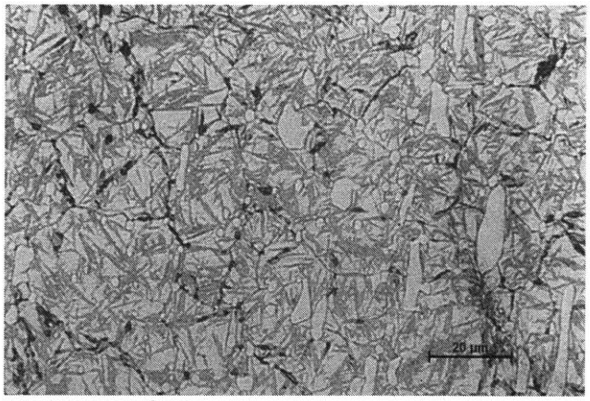
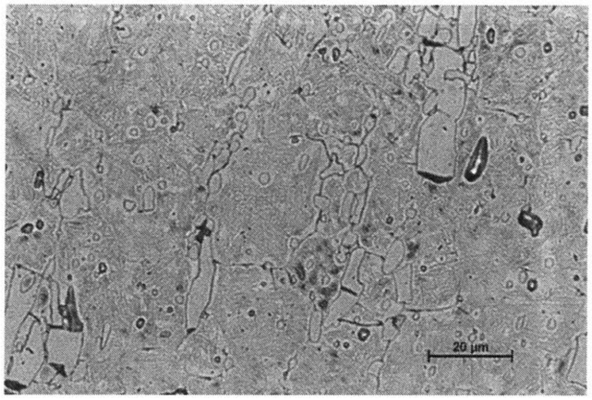
[42] (Verhoeven 2008, 24)



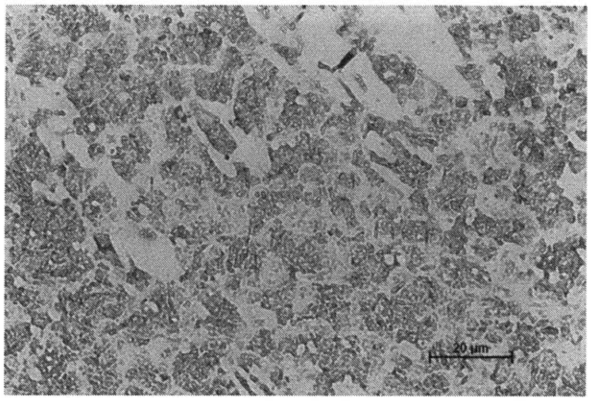
b) D2 Control



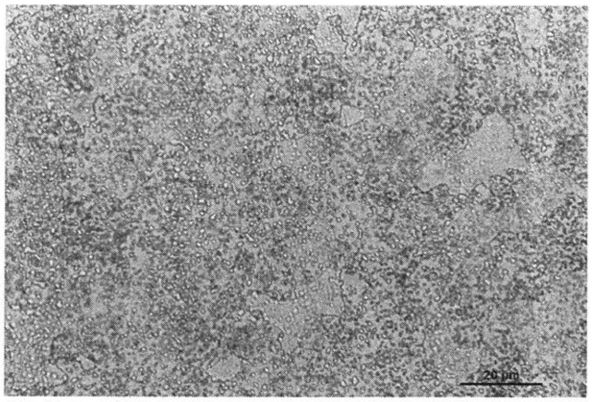
D2 Air Quenched



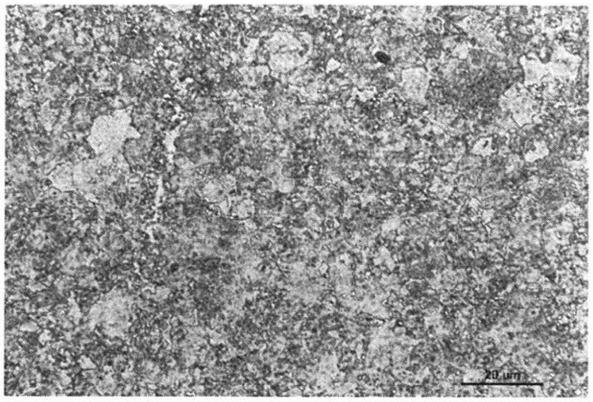
D2 Oil Quenched



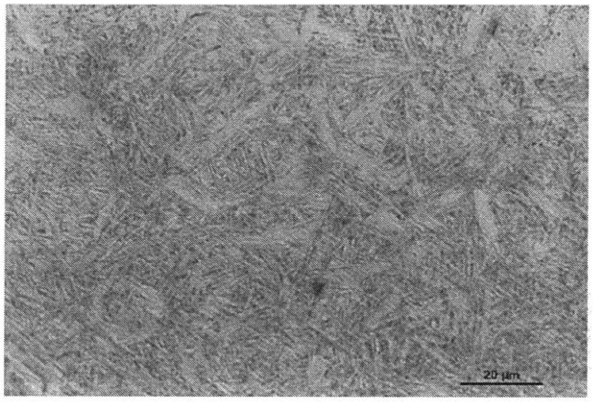
D2 Water Quenched



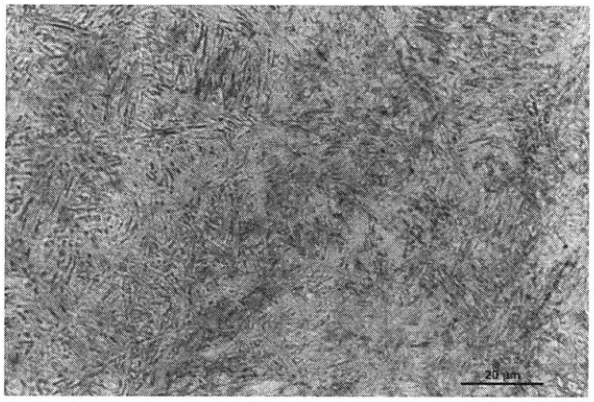
c) O1 Control



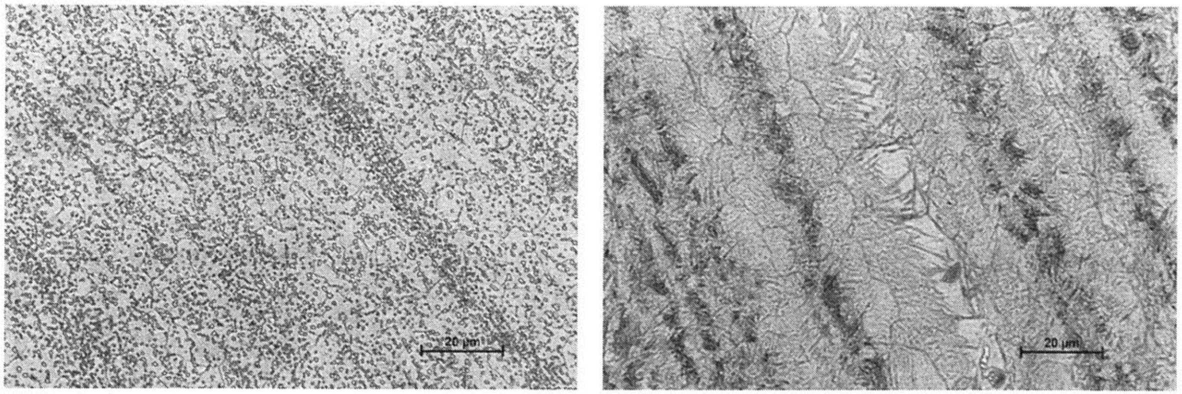
O1 Air Quench



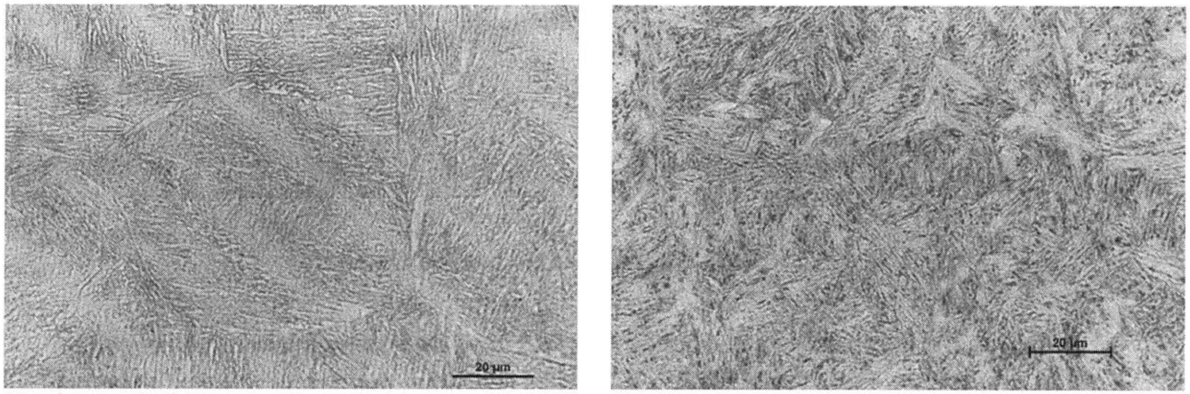
Oil Quenched



Water Quenched



d) S7 Control S7 Air Quenched



S7 Oil Quenched S7 Water Quenched

Figure 3-2: This figures displays the microstructure that was produced in four different steels under different quenching conditions. The images show the microstructure of 4140, D2, O1 and S7 steels respectively at 1000X magnification .The figures are arranged so that each of the top left images are the control samples, top right are air quenched, bottom left are oil quenched and bottom right are water quenched.

The temperature of the steel was recorded during the quenching process to visualize the cooling rate. The cooling data would be utilized with their respective TTT diagrams to understand how the steel's microstructure composition was determined. Figure 3-3 allows us to view the cooling process of water quenched steels, noting that the cooling time between 1200 Celsius and 84 Celsius was 8.73 seconds, fast enough to avoid the formation of any other phases such as pearlite, bainite or cementite. This observation is true for all the water quenched materials as the microstructure of all water quenched samples consist of martensite. Figure 3-3, shows the cooling process of quenching in transmission oil, graphing the change from 1200 Celsius to 200 Celsius in about 32 seconds; the oil quench cooling rate is able to avoid the cooling curves for the production of certain phases but is not able to produce full martensite in its microstructure. Similarly, air quenching is not able to produce martensite in its structure due to its very slow cooling rate which Figure 3-3, taking approximately 3.5 minutes to cool.

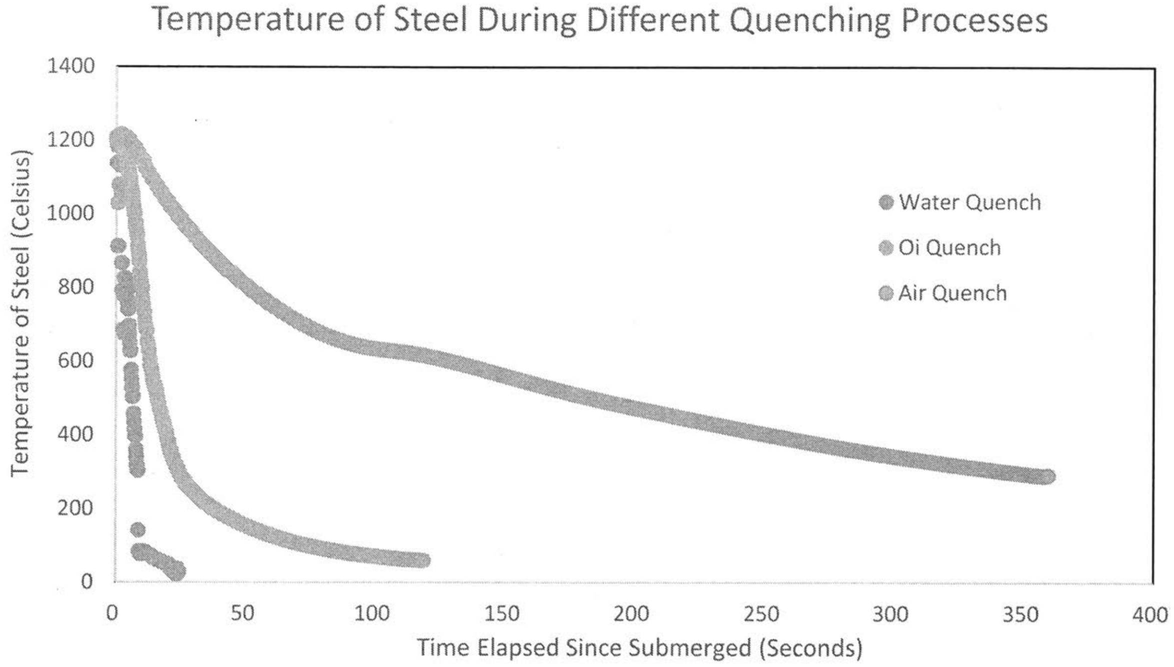
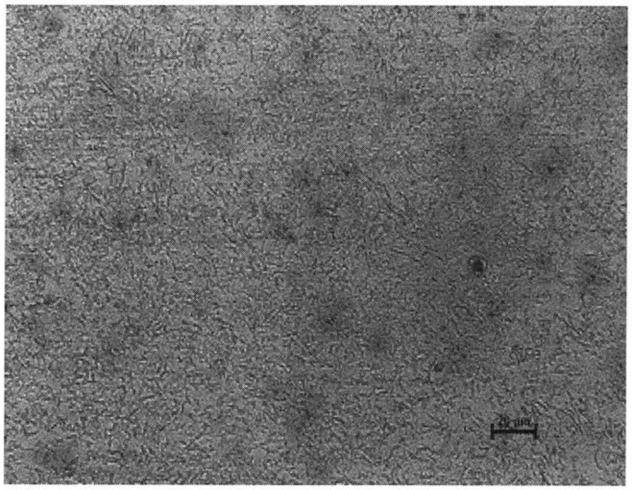
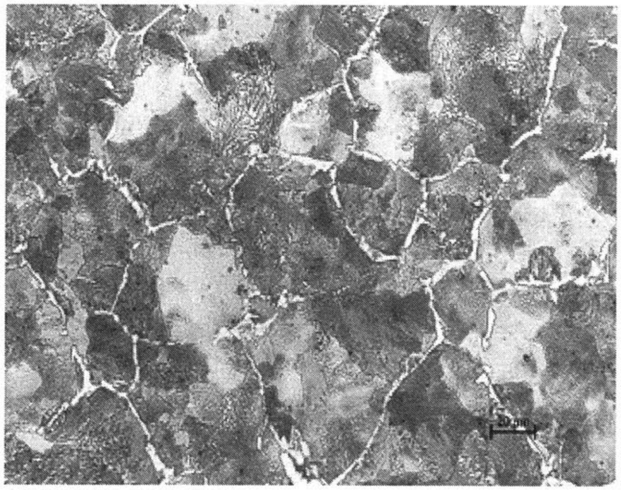


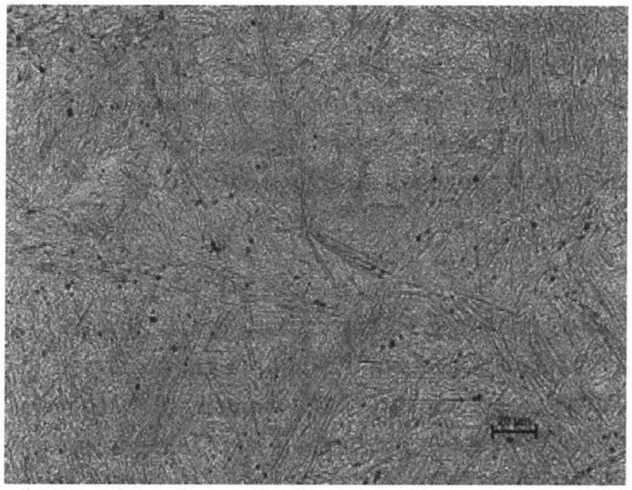
Figure 3-3: The graph shows the cooling rate that steel experiences when submerged in water, oil, and air. From the graph it is able to be seen that the temperature of the steel when submerged in water is able to be drop from 1200 Celsius to 200 Celsius within 8.73 seconds. Oil 1200 Celsius to 200 Celsius in about 32 seconds. Air took approximately 1 minute for the steel piece to reach 600 Celsius from 1200 Celsius, before its cooling rate slowed, taking approximately 3 minutes for the steel piece to cool from 600 Celsius to 300 Celsius.



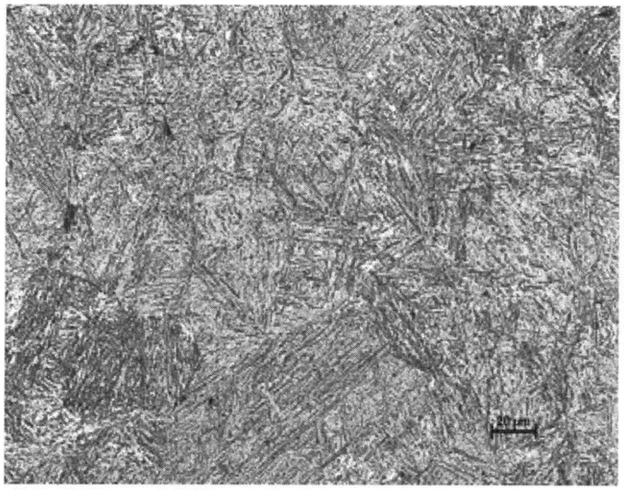
a) 1045 Control



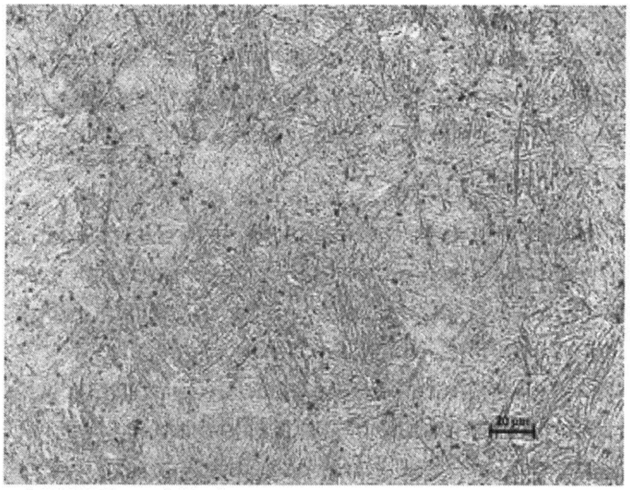
b) 1045 Air Quenched



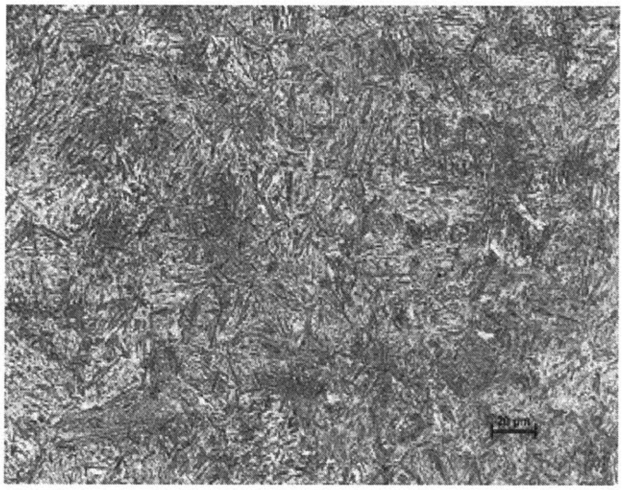
c) 1045 Olive Oil Quenched



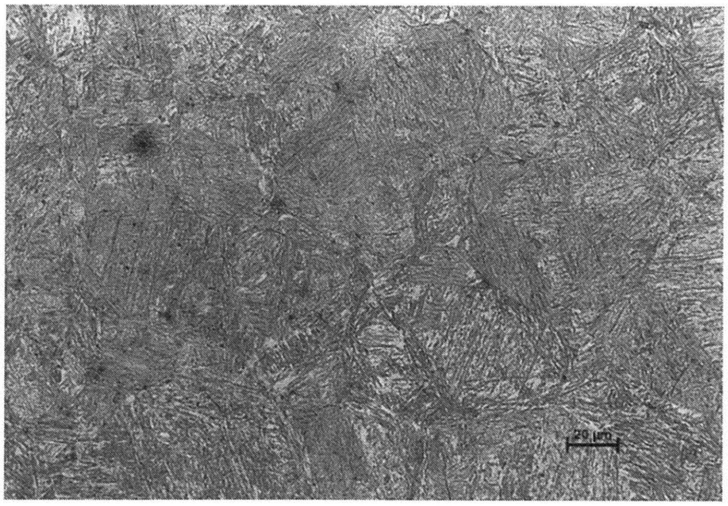
d) 1045 Peanut Oil Quenched



e) 1045 Quench Fast



f) 1045 Super Quench



g) 1045 Water Quenched

Figure 3-4: The images show the different microstructures that were observed in 1045 steel that include control samples and different quenching methods. In alphabetical order, the microstructures observed are from control samples and quenched samples from the mediums of olive oil, peanut oil, Quench Fast, Super Quench and Water. The samples are at 1000X magnification and show that all quenched samples except for air quench consist of mainly martensite.

3.2 Unconventional Quenching Methods

Due to the low variation in hardness, which can be viewed on Figure 3-5 through the graph, certain quenched pieces can be grouped. The oils such as Quench Fast, Olive, and Peanut are going to be grouped together for analysis. Looking at their microstructures it appears that a majority of them have martensite that has developed. This indicates that the cooling rate of the metal is were fast enough to avoid the complete formation of other phases such as ferrite, pearlite, and cementite. Looking closer at the values of hardness of the quenched samples by using Table 4, there is variation in the group of oils with Peanut Oil having a lower hardness but a greater standard deviation compared to Quench Fast and Olive oil who have a higher hardness and lower standard deviation. The observed microstructure in the steel samples do not match the predicted outcome from comparing the recorded cooling rates and the TTT diagram. The oil according to the TTT diagram cools too slowly to produce martensite in its microstructure, yet the microstructure shows mainly martensite.

Air quenched and control steel pieces will be grouped together due to their similarities in microstructure and hardness. Table 4 shows that Air quenched 1045 provides a harder material compared to the control steel sample. In the control piece there has cyclic heating that has allowed for the formation of spheroidization in the steel that makes the piece easier to machine, which can be viewed in the microstructure as consistent granules. However, in the air quenched piece the microstructure consists of large grains that are inconsistent in size with a white phase in the middle of the grains meaning that either ferrite or cementite were forming in those boundaries. The large grains of pearlite, ferrite, and cementite contribute to the air quench sample having a lower hardness than other oil quenched samples. The air cooling rate chart from Figure 3-3 compared to the TTT diagram of 1045 suggests that the cooling rate leads to the formation of pearlite and the development of larger grains that come from the low cooling rate.

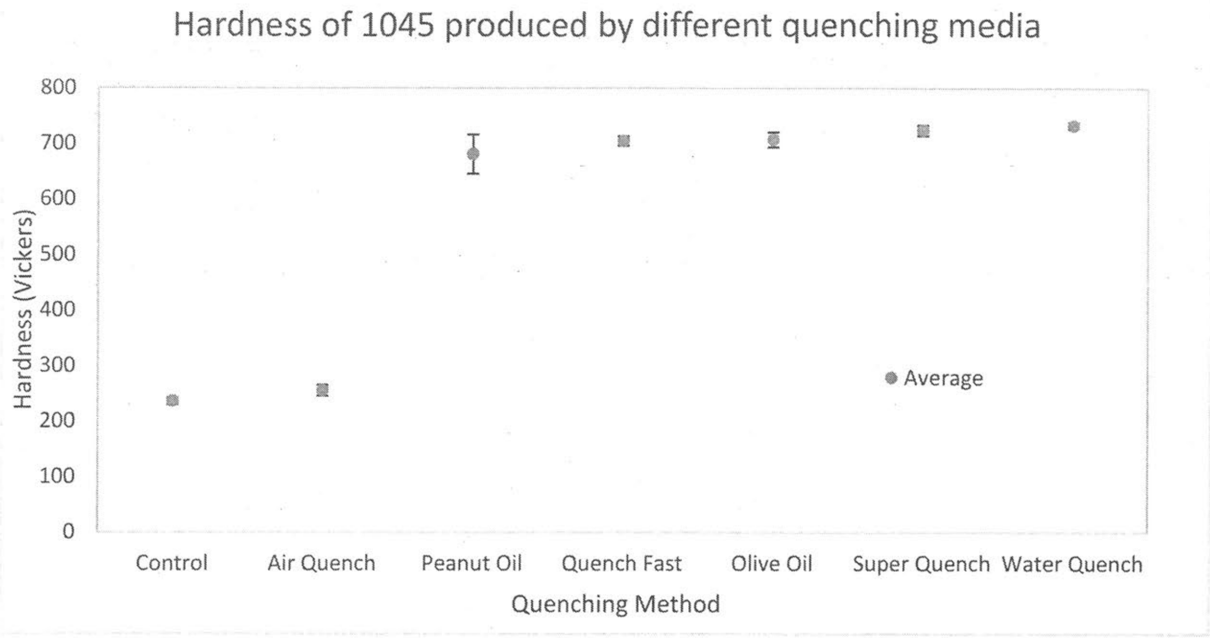


Figure 3-5: The graph displays the average hardness of 1045 steel after undergoing heat treatment and being quenched in different media with the standard deviations being represented as error bars. We can see that the control and air quench methods have similar hardness. Additionally, it can be seen that the oils, water, and unconventional quenching methods have similar hardness, within the range of 50 Vickers Hardness. The standard deviation of each quenching method is lower than 10 Vickers Hardness for all samples except for Peanut Oil Quenching which has a standard deviation of 30 Vickers Hardness.

Hardness of 1045 Steel After Different Quenching Methods on 1045 Steel (Vickers)

	Edge	Middle	Center	Average	Standard Deviation
As Received (Hot Rolled)	241	238	229	236.0	6.2
Air Quench	263	259	244	255.3	10.0
Peanut Oil	706	697	641	681.3	35.2
Quench Fast	713	706	696	705.0	8.5
Olive Oil	692	717	713	707.3	13.4
Super Quench	720	717	735	724.0	9.6
Water Quench	728	739	732	733.0	5.6

Table 3-2: The table shows the Vickers Hardness of steel samples after they have been heat treated and quenched in different media in three different locations of the sample. The varying steel hardness have been averaged and their standard deviation calculated.

Looking at the values between Super Quench and Water Quench there is little to no significant difference in hardness between the two media. From the data we took, Super Quench had a lower hardness than water by 5 which can be attributed to the standard deviation that was present in the sample data we took. Additionally, inspecting the microstructure of the two samples with quenching media of Super Quench and Water Quench, we can see martensite being developed throughout the piece evenly and very similarly to each other.

4. Conclusion:

When looking at the initial steel quenched samples that were prepped with different steels and conventional quenching methods we can see that there is a clear increase in the hardness of the steels. The increase in hardness can be concluded that it comes from the change in media as its specific heat rises. This increase in hardness correlates with discussions which state that with higher cooling rates there is an increase in hardness in the steel due to the faster production of martensite. Furthermore looking at the microstructures that were present in the samples we can see the difference between the conventional quenching methods. Water produces a microstructure of martensite in all samples, oil quenching produces a mixture of martensite and other phases such as pearlite and ferrite, and air quenching produces large grains that are a mixture of ferrite, pearlite and cementite. The observed microstructures in air and water correlate with the expected microstructure when comparing the measured cooling rates and 1045's TTT diagram. However, oil produces a microstructure in 1045 that was not expected when the TTT diagram and the temperature of steel during oil quenching were examined together. Oil was expected to produce weaker phases in the microstructure due to its slow cooling rate instead it was able to produce mainly martensite in its structure.

In the further study the effects of unconventional quenching media on hardness and microstructure with 1045 steel were observed to view the difference between vegetable oils, transmission oils, super quench, quench fast, and water. There is a distinct difference between using air and liquid as the quenching media in the microstructure and hardness with air quenching having a softer material property and a microstructure that reflects the softer composition.

Super Quench and Quench Fast, two unconventional quenching media, had minimal and indistinguishable variance to water or other oils respectively in 1045 steel. Water quenching was able to result in higher hardness than Super Quench, although the hardness of the two quenching methods were within the standard deviation of each method's sample data. The measured hardness of the water and Super Quench quenching media were within 5 Vickers Hardness of each other. Super Quench advertised having a higher specific heat and negating the Leidenfrost effect due to the soap and jet dry in online forums which would allow for a faster and more even cooling. Super Quench's effect on the 1045 steel was similar and almost undistinguishable when compared to the water quenched sample when inspecting the produced microstructure observing a microstructure composed of mainly martensite. The similarities in the hardness and microstructure of the Super Quench and water quenched samples might be due to specific heat of the solutions being similar to each other as Super Quench's base is mainly water. The similarity of specific heat would contribute to having a similar cooling rate which would contribute in the formation of martensite and would also in the evasion of the cooling curves of ferrite, pearlite and cementite formation. Therefore, it can be said that there is no advantageous reason to use Super Quench rather than water with 1045 steel for material hardness as both quenching process produce hardness and microstructure that are very similar with hardness that are within the standard deviation of each other.

On closer inspection, there is a more distinct difference between the oil group, and the water and Super Quench group. The water and Super Quench group produce hardness that is greater than the oils, but inside that group the hardness and microstructure varies little. The hardness in the oil group has a greater variance with peanut oil having the lowest hardness at about 20 Vickers Hardness lower than Quench Fast and Olive Oil. Peanut Oil having the lowest recorded hardness of the oil quenches is surprising as it has the highest specific heat meaning that it should have had the highest heat flux and should have corresponded with a higher hardness than the other media. Quench Fast and Olive Oil have comparable hardness averaging out at 705 and 707 Vickers Hardness respectively. Additionally the microstructure that Quench Fast and Olive Oil have are very similar to each other consisting of evenly distributed martensite throughout the sample. From the oil quench methods, Olive Oil and Quench Fast are able to produce similar end products meaning that Olive Oil can be used as a cheaper and more sustainable substitution for Quench Fast when needing to obtain similar hardness and material properties in 1045 steel.

We can make the conclusion that with 1045 steel, taking the extra steps to create a quenchant that should have a higher specific heat and properties that would avoid the development of a vapor blanket around the steel is not beneficial or advantageous against using water when both pieces that are being quenched are being treated with agitation. Additionally we can make the assumption that between Quench Fast and Olive Oil there is little difference in the product of the heat treatment as the hardness is within 2 Vickers Hardness units of each other, and the microstructure of both of the steels seem similar in composition. These results mean that using Quench Fast and Super Quench which have been advertised on forums for the rapid quenching properties are not qualitative improvements, and using quenchants such as Olive Oil and Water produce steels in similar quality and material properties with less preparation and improved sustainability.

These conclusions are not suitable to be applied to all steel grades or all kinds of applications. High quenching rates can lead to distortion and cracking of the steel whose effects were not included in this study.

5. References

Adebayo, A, Stephen, J., and G. Adeyemi. 2018. " Effects of Local Cooling Media on the Mechanical Properties of Heat Treated Mild Steel." *European Journal of Engineering Research and Science. 3 and 4* 27-31.

Adekunle, A., K. Adebiyi, and M. Durowoju. 2013. "Impact of quench severity and hardness on AISI 4137 using eco-friendly quenchants as industrial heat treatment." *Journal of Mechanical Engineering and Sciences* 409-417.

Brown, Dave, and Gunter Robb. n.d. *SOAP SOLUTION QUENCH: For Mild and Low Carbon Steels.* Accessed December 15, 2018. magichammer.freeservers.com/robb_gunter.htm.

Engineering ToolBox. 2003. *Specific Heat of Liquids and Fluids.* Accessed December 18, 2018. https://www.engineeringtoolbox.com/specific-heat-fluids-d_151.html.

KORAD, Thanaporn, Mana POLBOON, Niphon CHUMCHERY, and John PEARCE. 2011. "Improving Quench Hardening of Low Carbon Steel." *Journal of Metals, Materials and Minerals, Vol.21 No.1* pp.67-74.

Shinde, Rajkumar, and Dinesh Rao. 2013. "Influence of Petroleum and Biodegradable Quenchants on Properties of Medium Carbon Steels." *International Journal of Science, Engineering and Technology Research 2, no. 11,* 2052-2056.

University of Cambridge. n.d. *Interpretation of the Microstructure of Steels.* Accessed December 15, 2018. https://www.phase-trans.msm.cam.ac.uk/2008/Steel_Microstructure/SM.html.

Verhoeven, John D. 2008. *Steel Metallurgy for the Non-Metallurgist.* Ohio: ASM International.