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Characterising the Effects of Heat Treatment on 3CR12 and AISI 316 Stainless Steels

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Abstract**—**This paper reports on the effects of heat treatment on 3CR12 and AISI 316 stainless steel grades. Heat treatment was conducted on the steel grades and cooled using two different media; air and water in order to study the effect of each medium on the evolving properties of the samples. The heat treated samples were characterized through the evolving microstructure and hardness. It was found that there was a significant grain size reduction in both the heat treated stainless steel specimens compared to the parent materials. The finer grain sizes were achieved as a result of impediment to growth of one phase by the other. The Vickers microhardness values of the heat treated samples were higher compared to the parent materials due to the fact that each of the steel grades had a proportion of martensitic structures in their microstructures thereby improving the integrity of the material.

Keywords**—**Austenite, Ferrite, Grain size, Hardness, Martensite, Microstructure and stainless steel.

I. Introduction

THE continuous search for higher efficiency and productivity across the whole spectrum of manufacturing and engineering industries has warranted that most recent components are exposed to progressively severe environments during routine operation. Critical industrial components are as a result, susceptible to rapid degradation as the parts fail to resist the severities of destructive operating conditions and this has had a huge impact on the industry's economy. In most cases, the fast deterioration of components and their subsequent failure have been found from material damage brought about by unfavorable environmental conditions and by corrosive substances, cyclic stresses and extreme temperatures. Acknowledging that a large number of engineering parts fail catastrophically during service, through corrosion related occurrences, has further promoted this approach and led to the advancement of the stainless steels modification methods using the appropriate heat treatments. Stainless steels represent the most diverse yet complex family of all steels. Stainless steel is a part of a large group of alloys with different properties and consists of a spontaneous layer which forms on their surface and diminishes the rate of corrosion to almost insignificant levels [1]. Globally, the demand for stainless steels is increasing at a rate of approximately 5% per annum [1]. Annually, over twenty million tons of steel is consumed and this occurs mainly in the construction industry and household appliances. New uses are constantly being found for the attractive appearance, corrosion resistance, low maintenance and good strength. Stainless steel costs more compared to the standard grades of steel because of its low maintenance and have no need for protective surface coatings or painting. The aforementioned factors thus make stainless steel more economically viable when the service life and life cycle costs are considered. Based on their crystalline structure, stainless steels are classified into three basic groups such as austenitic, ferritic and martensitic. Ferritic stainless steels such as 3CR12 are emerging as an interesting and cost effective alternative to more costly materials in many applications. This material is created to be utilized for automotive exhausts, fuel lines, and water tanks, cooking utensils or even architectural members carrying loads [2]. Nonetheless, very little information devoted exclusively to ferritic grades is available in the literature as opposed to austenitic steels which are covered immensely. Austenitic stainless steels such as AISI 316 are distinguished by their suitable applicative nature due to their good combination of high chemical properties. These properties are dependent and influenced by quantity and nature of their alloying elements. They are also dependent on the heat treatment used. It is expected that by subjecting both steels to heat treatment, the structure of the metals is modified to be beneficial for industrial use and meet structural requirements in practical applications. Therefore, the present work attempts to provide a further understanding of the effects of heat treatment of AISI 316 and 3CR12 stainless steel grades on the evolving microstructure, hardness and grain sizes. The effect of the heat treatment process on stainless steels is necessary in order to be able to distinguish between the mechanical as well as the metallurgical properties of the steels in their as-cast state and heat treated state. For the past decades, heat treatment methods have been developed through a number of substantial experiments and characterization of certain alloy compositions and final properties [2], [3]. This process, together with an extensive experimental database frequently accomplishes the required properties however, the process is limited in that it cannot be altered to meet the increasing demand for quality and cost optimization and to meet the strict standards as well as not being able to be extended to newly developed steels. In this regard, techniques which enable the prediction of the evolution of microstructure as a function of the composition and the heat treatment cooling media are very beneficial. Studies in this field of research include the study conducted by

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Knutsen [4]. The microstructural evolution of 3CR12 steel during cooling from a 1380℃ to 25℃ solution heat treatment was conducted and characterized. The microstructural analysis revealed the development of a banded, dual phased microstructure of martensite and ferrite. In this formation, a reduction of Chromium and enhancement of Manganese and Nickel in the martensite were discovered. In a similar but separate study by Hewitt [5], it was observed that for low cooling rates (1℃/min) of 3CR12 austenite transforms fully to ferrites plus carbides without any traces of martensite. And at intermediate cooling (less than 60℃/min), the austenite transforms to martensite and ferrite plus carbides. It was noted that the cooling rate directly influences the ratio of ferrite to martensite in the heat treated 3CR12 [5].

In an investigation on titanium stabilized 3CR12, Ball et al., [6] for heat treating temperatures of below 700℃, the austenite transformed fully into ferrite with carbides at a time of 14 hours and the ferrite grew epitaxially on the pre-existing δ-ferrite. At a temperature of 1100℃, the final microstructure of the heat treated sample of 3CR12 was martensite and ferrite plus inter-phase precipitation of carbides.

Also, in a study by Kciuk and Kurc-Lisiecka [7] to investigate the influence of heat treatment on the structure, mechanical properties and corrosion resistance of X10CrNi18-8 steel, the microstructure of the samples annealed at 1050℃ was characterized by austenitic single phase structure, the annealed samples had a hardness value of 375 HV, it was also noticed that when the temperature was increased, the hardness value was decreased to 203 HV. The structure of the steel in the as-received conditions consisted of austenitic microstructure with numerous slip bands in areas with deformation martensite.

An investigation by Bayram et al., [8] on 0.1% carbon steel which was subjected to three different kinds of heat treatment yielded the following; the inter-critically annealed steel portrayed an equi-axed ferrite-martensite structure which exhibited the highest tensile strength and lowest ductility. The step quenched steel also yielded a ferrite-martensite structure with the worst mechanical properties of the three heat-treatment methods. In contrast to the other two heat treatment methods, step annealing yielded a fibrous (fine, needle-like) ferritic-martensitic structure.

In a study to determine the influence of annealing on microstructure and hardness of 3CR12 steel by Ulvan et al., [9] the microstructure of the as-cast 3CR12 was found to consist of martensitic grains in a matrix of delta ferrite. The steel samples were annealed at 725℃ for 25 minutes. The hardness of the ferrite and martensite in the 3CR12 steel was measured and the overall hardness of the material was determined and found to be 195 HV.

In a research study conducted by Blum et al., [10] to determine the influence of microstructural parameters on the yield stress and fracture toughness of 3CR12 steel, it was observed that the yield stress of the as-received 3CR12 steel, exhibiting a banded dual-phase microstructure, was much lower than that of the flake-like dual-phase microstructure created by heat treatment. The yield stress of the dual-phase 3CR12 steel in the heat-treated state was found to be considerably higher than that in the as-received condition. The increase was attributed to changes in the microstructure that give rise to different constraint effects under which the ferrite phase deforms [10].

In a separate study, Ganesan and Ganesan [11] investigated the properties of solution annealed AISI 316 austenitic stainless steel specimens exposed to static sodium at 873℃ for 500 hours. The results of the hardness values and microstructures of the fully annealed specimens were obtained. The microhardness measurements showed an increase in hardness values from 150 HV in the annealed specimens to 250 HV after sodium exposure at 873℃ for 500 hours. The average grain size was determined as 50μm.

A theoretical and experimental study was conducted by Di Schino and Kenny [12] on the recrystallization and grain growth processes on AISI 316 stainless steel. The steel was annealed at a temperature of 1100℃ for up to 8 minutes. The samples were analyzed using automatic image analysis to observe the resulting micrographs. Microhardness measurements were also taken on the samples and related to the recrystallized volume fraction using a mathematical model based on statistical assumptions capable of defining recrystallization and grain growth in metals. The average grain size was between 10-30μm. In order to evaluate annealed samples of AISI 316 stainless steel, a microstructural analysis was also performed by Solomon and Solomon [13]. The 316 samples highlighted an austenitic structure with annealing twins and some non-metallic inclusions which were identified as oxides, the average grain size on all tested the samples was 7.4μm. This then means that the austenite stability decreases with increasing grain size. Hence, coarse-grained austenite is more susceptible to martensite transformation. It was also found that the corrosion resistance is influenced by the amount of martensite formed during the steel heat treatment. Austenitic steels are used in varying industrial applications and hence it is important to control their microstructural evolution, physical properties and mechanical properties sinceplastic deformation causes the transformation of austenite into martensite for these types of steels.

A study was also done by Long [14] in order to identify the effect of quenching process bases on hardenability, microstructure and distortion profiles of three different stainless steels one of which was AISI 316. Three different experiments were conducted which included hardness testing, microstructural analysis and distortion examinations. The samples hardness profiles across the bars showed a hardness decrease toward the centre. The sample with the fast cooling rate (water quenched) transformed from an austenitic phase into a new martensitic phase. The martensite looked needle-like under the microscope with a fine lamellar structure. After quenching, the sample was hard but brittle. It was concluded that the quenched sample had a high hardness profile. This was seen after all samples with different quenching media were observed. Thus the faster the cooling rate, the harder the resulting structure [14].

In a related study, Ramanathan and Foley [15] carried out experiments to determine the evolving microstructure of nine different steels. The steels were heated in a furnace to a temperature of 905℃ and thereafter some were air cooled and others oil-quenched to produce three different microstructures. Amongst the heat treated steels was austenitic stainless steel AISI 316. This steel revealed almost the same microstructure under all heat treatment conditions. The authors observed that the matrix of this austenitic stainless steel does not experience phase transformation. The most notable significant finding was the fact that the microstructure indicated that the grains were refined as the cooling rate progressed from air-cooling to oil-quenching [15]. In a recent study [16], the effect of heat treatment on transformation induced plasticity of Cr19 duplex stainless steel was conducted by Ran et al. The effect of the annealing recrystallization treatment from 950°C to 1250°C for 3 minutes and isothermal aging treatment at 750°C for one to seven hours on transformation induced plasticity was conducted and it was found that all the annealed specimens exhibited moderate strain hardening but became less obvious with the increase of the annealing temperature. Heat treating stainless steels such as 3CR12 and 316 will enable restoration of desirable properties such as corrosion resistance to metal altered by prior manufacturing operations or produce hard structures able to withstand high stresses or abrasion in service. The reviewed literature indicates that the mechanical and metallurgical properties of stainless steel grades AISI 316 and 3CR12 can be manipulated by using heat treatment methods applicable to each steel type. Several authors have demonstrated similar findings regarding the heat treated 3CR12 and AISI 316, particularly on the microstructural analysis and the microhardness values. From the various results obtained, it can be concluded that the mechanical properties vary depending upon the various heat treatment processes employed. There is very little information on the relationship between using different quenching media and the hardness and grain sizes of these grades of stainless steels.

In view of this, the present study is aimed at investigating the effects of heat treatment on the evolving microstructure, hardness and the grain sizes of stainless steels, specifically the 3CR12 and 316 steel grades by using different cooling media.

II. Experimental Set Up

The parent materials used in this research work were AISI 316 and 3CR12 stainless steels. The dimensions of the test specimen for each sheet were 100 x 100 x 3mm^3. The as-received material; AISI 316 and 3CR12 steel samples were heat treated at a constant temperature of 1200℃ and each was quenched either in water or air cooled in order to modify the metallurgical and the mechanical properties. A Lenton Furnace shown in Fig. 1 at the Foundry of University of Johannesburg was used for heat treating the stainless steel samples.

Fig. 1 The Lenton Furnace

The furnace was switched on and pre-heated to a temperature of 1200℃. The samples of AISI 316 and 3CR12 stainless steels were placed in the pre-heated furnace for a period of 12 hours.

After the heat treatment process, a sample of the AISI 316 steel was removed using a pair of tongs and thereafter quenched in a water solution and another sample was placed on a heat resistant block and allowed to cool off in air at room temperature outside the furnace. A sample of 3CR12 was also removed from the furnace and water quenched while another was cooled by air at room temperature. The samples were then sectioned, mounted in hot polyfast resin and prepared in accordance with ASTM standard [17]. The samples of 3CR12 were etched using a solution of 50ml ethyl alcohol and 50ml of hydrochloric acid while the AISI 316 samples were etched with aqueous oxalic acid. The etched samples were observed under the optical microscope (BX51M). The Zwick Roell Vickers hardness tester was used to measure the microhardness. The Vickers hardness was measured with a load of 400g force, and the dwell time was 15 seconds. The microhardness profiles were conducted with a 0.1 mm interval between each indentation. The sample labels and designations are as presented in Table I.

TABLE I
Sample Labels and Designation

Sample		AISI 316	3CR12
Parent material	PM	A1	A2
Heat treated and aircooled	WQ	B1	B2
Heat treated and water quenched	AC	C1	C2

The samples were labeled as presented in Table I for easy identification during the course of the research work.

III. Results and Discussion

A. Microstructural Evaluation

The microstructures of the parent materials of both the 3CR12and AISI 316 steels, the annealed and water quenched 3CR12 sample, the annealed and air cooled 3CR12 sample, the annealed and water quenched 316 and lastly the annealed and air cooled 316 steel samples are presented in Figs. 2 (a)–

(f) respectively.

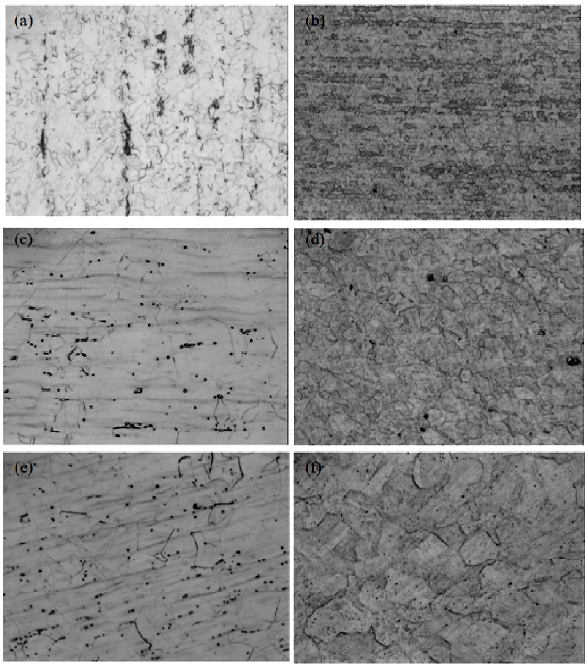

Fig. 2 Microstructures of (a) As-received AISI 316(b) As-received 3CR12 (c) Heat treated and water quenched AISI 316 (d) Heat treated and water quenched 3CR12 (e) Heat treated air cooled AISI 316 and (f) Heat treated air cooled 3CR12

Prior to the heat treatment, the microstructure of the as-received AISI 316 specimen Fig. 2 (a) reveals the existence of a minor ferrite phase (dark phase) in a dominantly austenite phase structure (white phase); these are consistent with a 316 stainless steel alloy; the grainy and lamellar structures are ferrite while the as-received parent material of 3CR12 stainless steel grade (Fig. 2 (b)) is seen to have a ferrite-martensite appearance. The ferrite appears light while the martensite phase is dark. The dual-phase banded structure consists of pancake shaped martensite grains separated by ferrite bands which lie in the rolling plane which is similar to the report of Knutsen [4]. The microstructure of the parent material of 3CR12 appears to have grains aligned in the longitudinal direction.

The micrograph of the annealed and water quenched AISI 316 as shown in Fig. 2 (c) shows grains that are mostly martensitic with minor presence of the ferrite phase. The volume of the martensite is seen to have increased significantly in this microstructure which is in agreement with [12]. The microstructure of the heat treated and water quenched 3CR12 specimen (Fig. 2 (d)), illustrates that the coarse ferrite grains in the parent material are transformed into a very fine duplex structure of ferrite and martensite due to the rapid cooling rate offered by water as a quenching medium. In addition, the minor dark contrast can be observed, indicating that carbides are in the solution. The grain size is also noted to be more refined compared to the air cooled specimen.

Fig. 2 (e), the microstructure of the annealed and air cooled AISI 316 stainless steel shows a dual-phase of ferrite-martensite. It can be seen that the microstructure shows very little forms of ferrite grains and only a homogeneous austenite microstructure with equal grains and the presence of annealing twins is observed similar to the report of Solomon et al., [13] while the micrograph of the heat treated and air cooled 3CR12 (Fig. 2 (f)) presents grains that are fairly rounded polygons. The heat treated and air cooled 3CR12 produced a coarse dual phase microstructure. The annealed sample shows equi-axed grains of ferrite and globular precipitates of carbides as shown. The findings are consistent with [4], who also reported partitioning of Chromium to the ferrite and of Nickel and Manganese to the martensite in 3CR12 steel after heat treatment of stainless steel 3CR12.

B. Grain Size Characterization

The observed grain deformation which has occurred in the evolving microstructures of the heat treated steels was quantified and characterized. The measurement tool on the optical microscope was used to measure the size of the individual grains in the microstructures. The percentage decrease in the grain size was determined with reference to the average grain size of the parent material. The average grain size was determined by measuring five individual grains in each steel sample. The data is provided in Table II.

TABLE II
GRAIN SIZE DATA

Specimen (Grain)	316 (A1)	316 (B1)	316 (C1)	3CR12 (A2)	3CR12 (B2)	3CR12 (C2)
1	42.2	22.5	25.1	40.2	25.2	10.5
2	55.0	24.6	22.4	43.1	23.1	11.0
3	33.4	18.7	16.0	34.5	24.0	18.0
4	32.2	29.9	18.0	35.5	23.8	14.2
5	40.5	20.5	14.9	25.8	23.5	10.7
Average grain size (µm)	40.7	23.2	19.3	35.8	23.9	12.9
%decrease in grain size compared to parent material		42.3	52.6		33.2	64.0

The degree of grain deformation in the AISI 316 and 3CR12 stainless steels are presented pictorially in Figs. 3 (a) and (b) respectively.

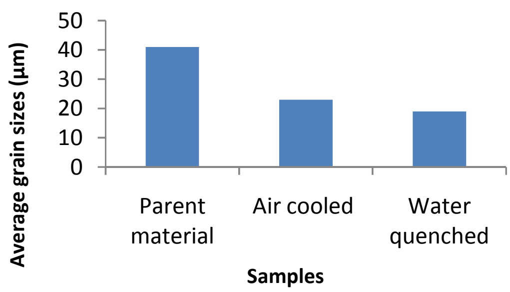

Fig. 3 (a) Average grain sizes of AISI 316 as-received and the heat treated samples

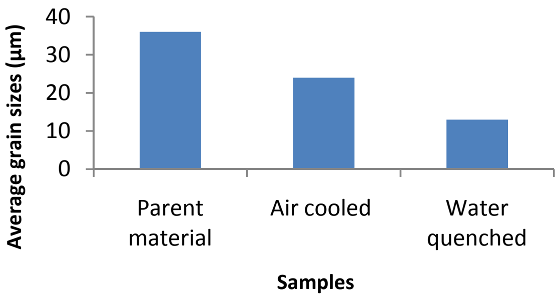

Fig. 3 (b) Average grain sizes of 3CR12 as-received and the heat treated samples

The general trend which can be seen is the significant reduction in the grain size from the parent material to the heat treated specimens. The water quenched specimen grain size was reduced drastically compared to the air cooled specimen. The notable observations are that the heat treatment process reduced the grain sizes of the AISI 316 and 3CR12 stainless steels. Also, it is implied that the air cooled specimens still had coarser grain sizes whereas the water quenched samples had a more refined grain size. The Hall-Petch relationship seemingly exists between the fine and medium grain size specimens, but the coarse grain specimen may have deviated from the Hall-Petch relationship due to too few grains in the specimen cross section.

From the tabulated results in Table II, the reduction in grain size of the heat treated 3CR12 samples was found to be 33.2% for the air cooled specimen and 64% for the water quenched specimen. The reduction in grain size for the heat treated AISI 316 specimens was found to be 42.3% in the air cooled sample and 52.6% in the water quenched sample. This is attributed to the process of recrystallization that has occurred during the annealing process.

In the course of recrystallization, the formation of a new grain structure by the growth of new grains from nuclei is developed. Fewer dislocations are induced in the material during air cooling so that less nucleation sites are available for the growth of new small grains. As a result, fewer grains are formed during recrystallization, which grow until they touch each other so that there is a larger grain size. The opposite occurs during water quenching as more dislocations are induced into the material followed by many nucleation sites. Hence, more new grains can grow but they will not grow as much because they touch each other after a short time as there are many new grains. Therefore, the water quenched specimen has the smallest grain size compared to the air cooled specimen.

C. Vickers Microhardness Profiling

The Vickers microhardness profiles of the as-received AISI 316 and the 3CR12 stainless steels and the heat treated samples are presented in Figs. 4 (a) and (b).

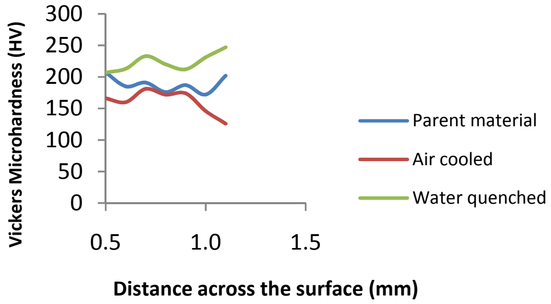

Fig. 4 (a) Vickers microhardness profile of AISI 316 as-received and heat treated samples

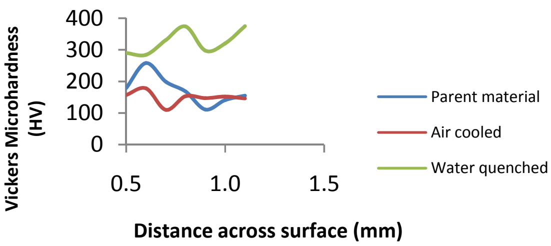

Fig. 4 (b) Vickers microhardness profile of 3CR12 as-received and heat treated samples

The base material of AISI 316 showed an average hardness of 160 HV which correlates to the typical hardness value of as-cast AISI 316 steel grade of approximately140 HV. The heat treated and air cooled specimen exhibited an average microhardness of 188 HV which was lower compared to that of the heat treated and water quenched specimen at a microhardness of 223 HV while for the 3CR12, the average Vickers microhardness value of the parent material 3CR12 is 146 HV. This value is in correlation with literature [4] which suggests that as-cast 3CR12 rolled sheets have a microhardness value of ±150 HV. The heat treated and water quenched specimen developed an average microhardness of 324 HV which is much higher than the average microhardness of heat treated and air cooled specimen of 173 HV. The increase in hardness was expected as heat treating stainless steels for long periods of time results in a hard and brittle structure.

As observed in Figs. 4 (a) and (b), the microhardness profiles of the heat treated specimens increased compared to that of the parent materials. It can be deduced that the increase in the microhardness is due to the delay in the formation of the ferrite phase which in turn promotes the formation of the martensite phase during cooling. The microhardness of the water-quenched samples is increased significantly compared to the hardness values obtained from the air cooled samples for both the 3CR12 and AISI 316 steels. This is due to the dominant presence of martensite in the water quenched samples. Martensite is known to be one of the most common strengthening phases in stainless steels. The microhardness increases in the heat treated and water quenched sample compared to the parent material because of the refinement of the primary phases after rapid cooling by water [18].

It is well known that water quenching creates a supersaturated solid solution and vacancies increase with carbon content in water quenched samples [18]. As a result the high hardness correlates with high resistance to slip and dislocation.

The microhardness values provide a relative idea of the hardness of 3CR12 as well as AISI 316 stainless steels. Although, these values illustrate changes in hardness on a microscopic scale, they are specific. For the 3CR12 stainless steel, the heat treating process followed by water quenching seemed to harden the material significantly. The hardness of the air cooled specimen did not increase considerably as compared to the water quenched sample. This suggests that the method of rapid cooling 3CR12 in water generates results in increased hardness as compared to using air as a cooling medium. The consistency of the HV values of 316 steel samples correlates with the obtained microstructures of the heat treated and the water quenched specimen. The equal sized grains were expected to illustrate a consistent microhardness values. The increase in the microhardness values for the heat treated specimens can be attributed to dynamic recrystallization that has occurred during heat treatment.

IV. Conclusion

The effects of heat treatment using different cooling media on AISI 316 and 3CR12 stainless steels have been presented. The microstructural evaluation revealed that heat treating 3CR12 results in a dual phase microstructure of martensite and ferrite. On the other hand, AISI 316 retained its austenitic phase throughout the heat treatment and this occurs because for austenitic steels, the range of temperatures at which phase transformation occurs is below 1200℃ and so above that range of transformation temperature the steel remains unchanged. The effect of heat treating stainless steels was quantified and characterized. Heat treating 3CR12 and AISI 316 with water as a quenching medium exhibited significantly higher hardness values as compared to the parent material. The air cooled samples of AISI 316 also exhibited higher hardness values when compared to the parent material. Moreover the water quenched samples showed higher HV values in comparison to the air cooled samples.

The grain sizes and the percentage reduction showed that heat treatment alters the grain sizes of the stainless steels. Furthermore, the reduction in the grain size of both stainless steels was significant. The grain sizes of both samples of 316 and 3CR12 decreased significantly. This is a desirable effect of the heat treatment on stainless steels. Hence, it can be said that the differences in the cooling media appear to have dramatic effects on the microstructure and the microhardness of stainless steels, the faster the steel was cooled, the harder it became and also the more the grain size was reduced thereby influencing the strength and the integrity of the heat treated samples.

Acknowledgment

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