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Investigation on the Interrelationship between the Chemical Composition, Heat Treatment Parameters and the Phase Transformation Process, Microstructure Evolution and the Mechanical Properties of Austempered Steel

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ABSTRACT: *The interrelationship between chemical composition, heat treatment parameters, and phase transformation, microstructural evolution and the mechanical properties of austempered steel was studied. Two samples of steel with different percentage composition of carbon of 0.56 and 0.76 were used for the study. They were austentized at the respective temperatures of 800^0C, 840^0C, 900^0C and 960^0C for 30 minutes. They were thereafter quenched using bitumen-palm kernel oil, and subjected to austempering isothermal heat treatment at $420^0C$for different time durations of 5, 15, 30, 45 and 60 minutes. The samples were tested for tensile strength, elongation, hardness and impact strength. They were also subjected to microstructural characterization to determine the phases in the microstructures and their effects on the properties of the developed materials. Results obtained revealed that the dominant phases in the microstructure were bainite, matensite and traces of retained austenite. It was found that decreasing austenitizing temperature yields finer grain structures with increase in tensile strength and elongation with decrease in hardness and relatively little effect on the impact strength. At any given austenitizing temperature, shorter austempering holding time yielded optimum properties in tensile strength and elongation while higher hardness values were associated with shorter holding time. These results proved that the process conditions have strong correlation with both the microstructures and the mechanical properties. It was concluded that the most promising microstructures with respect to excellent strength-ductility property are those obtained at the ausenitizing temperature range of 800 -840^0C for the austempering time range of 5 – 30 minutes. These materials have potential for load bearing application while those austenitized within the range of 900 - 960^0C using austempering time range of 5 – 15 minutes are candidate material for wear resistant application.*

KEY WORDS: Austempering, Microstructure, Heat treatment, Properties, Composition, Austenitize

INTRODUCTION

Steel is an alloy of iron and carbon in which the percentage composition of carbon ranges up to 2.14%. The presence of carbon in iron improves its strength and fracture resistance. Depending on the operation temperature, iron can take different crystalline forms in steel. It is the interaction of these allotropes with carbon that yields the structure in the alloy. The structure obtained determines the range of unique properties of the material. The changes in the micro structure could be achieved by adjusting the heat treatment process parameter and by the use of alloying elements other than carbon. [1].

Heat treatment of steel is a solid state transformation process which employs the use of regulated temperature control at various levels within a narrow range of compositions of the mixtures of carbon and iron to develop different metallurgical alloys with unique properties. Austempering is one of the heat treatment process applied to ferrous metal particularly to steel and ductile iron in order to improve the mechanical properties. The process is the same both in steel and cast iron. However, it produces baintic structure in steel while in cast iron, it yields a structure of acicular ferrite and high carbon, stabilized austenite known as ausferrite [2]**.**The design of austempering process in steel which produces bainitic structure yields properties that combine uniform and consistent hardness with toughness. This gives rise to high resistance to brittle fatigue. Other advantages of the bainitic structure include higher ductility, increased strength at a given hardness, increase toughness, greater fatigue life and less distortion. [2].

In order to achieve any transformation in austempering heat treatment in steel, the microstructure of the metal must be austenitic in structure. The initial process during the heat treatment process involves austenitizing at temperature ranges of790^0 C – 950^0 C.[3, 4]**.** This will help to facilitate transformation that yields austenite structure**.** The heat treated material is allowed at the same temperature for a specific time range in order to produce a full and uniform austenitic metal structure with consistent carbon content. [2]**.** The holding time is dependent on the nature of the alloy and the process undertaken. Quenching at a very fast cooling rate is necessary to prevent the formation of pearlite but should be above martensitic temperature so that the microstructure will be a of mixture of bainite and martensite. However, the dominating phase should be more of bainite. The severity of the quenching media depends on its ability to mediate heat transfer at the heat interface during quenching. The cooling rate and the holding time are important factors required to achieve the desired microstructure. [5, 6]**.**

The during austempering process such as temperature and time depends on the expected properties and the target area of application. Depending on the bainitic holding time, various mixtures of bainite, martensite and retained austenite can be found in the microstructure after quenching. [11]**.** Bainite morphology consist of aggregates of platelets of lath of ferrite, separated by region of residual phases of untransformed austenite or phases such as martensites or cementite which form subsequent to the growth of bainitic ferrite. The microstructure obtained during austempering process depends greatly on the process parameters and the composition of the alloy. [4]**.**These factors can affect nucleation and rate of grain growth. Many properties of steel such as tensile strength, yield strength, toughness and hardness depend on grain size**.** High transformation temperature requires less time to complete the bainitic reaction. When the isothermal holding time of steel is above eutectoid temperature for sufficient time, it ensures adequate diffusion of carbon atoms which results in a uniform distribution of carbon atoms. This yields a homogenous structure. [4]**.**

A lot of research studies have been carried out to determine the extent to which some of the heat treatment variable could affect the microstructure evolution of heat treated steel which in turn determines the material's properties. Ferrous alloys with different chemical composition respond differently to varied heat treatment parameters so as to yield various microstructure with specific mechanical properties. Therefore, it was concluded that the microstructure of metal determines its mechanical properties.[7]**.**

[4] Observed that varied mechanical properties could be obtained from two grades of steel having the same chemical composition and subjected to similar heat treatment. This suggests that some independent heat treatment factors could affect the phase constituents in the microstructure. In view of this, it was stated that, it is not only the presence of the phase micro constituents but also the morphology of these product that is significant in determining the resultant properties of heat treated steel. [8] Considered the effect of austempering temperature and time on the mechanical properties of SAE 9260 steel. From the results obtained, it was postulated that the volume fraction of retained austenite serves as a function of isothermal temperature and time hence impact toughness increases with increase in the volume fraction of retained austenite. [9] Studied on the effect of cooling rate on phase transformation of mild steel, they stated that acceleration on the transformation of austenite depends on the size of the resulting grain. [12] Used design of experiment to analyse the effect of quenching parameters such as austentization temperature, soaking time and cooling medium on hardness and the distortion of AISI 4340 steel. It was concluded from the result that the distortion increases with increasing austentizing temperature and time while cooling medium had significant effect on hardness. [17] Investigated the effect of austempering conditions on the microstructure and mechanical properties of AISI 4340 and AISI 4140 steel. From the findings made, it was established that decrease in austempering temperature increases the values of both hardness and toughness due to formation of large amount of fine grained lower bainite alongside martensite.

From these previous studies and their corresponding findings, it was observed that there is need to study the interrelationship between the production and process factors and the properties of austempered steel at different stages of the heat treatment. This will help to optimize the process conditions for the development of the candidate material required for specific application.

Therefore, the aim of this study is to enrich knowledge on the interrelationship between the chemical composition of heat treated steel, heat treatment variables and phase transformation process, microstructural evolution and the mechanical properties of austempered steel. The study will be a useful tool in process design for the development of various ferrous alloys that are suitable for many engineering applications.

MATERIAL AND METHODS

Materials

Two samples of steel (grade A and B) with the respective percentage carbon composition of 0.56% and 0.76% were used in this study. The chemical composition of the steel samples is shown in Table 1. A blend of 15:85 bitumen-palm kernel seed oil was used as a quenchant for the heat treatment process. The choice of the combining ratio was based on high oleic and linoleic acid content of palm kernel which has suitable characteristics of quenchant [16]. The equipment used include heat treatment furnace, lathe machine, grinding, universal tensile testing machine, Rockwell hardness testing machine (Model number 6186.5B) and metallurgical microscope.

Methods

Heat treatment process

Four groups of sample of each grade of steel (A and B) which were made up of five test samples were used in the study. The four groups of steel sample were austenitized at the respective temperatures of 800^0C, 840^0C, 900^0C and 960^0C for 30 minutes. The choice of the austentizing temperatures was based on the guide from the phase diagram shown in figure 1**.** This shows that the temperature range could guarantee complete formation and dissolution of austenite without allowing grain coarsening to set in. The steel samples were later subjected to austempering isothermal heat treatment where they were transferred from the elevated temperatures to a molten salt bath of bitumen-palm kernel mixture as quenchant.[10]**.** They were held at a constant austempering temperature of 420^0C for varied time durations of 5, 15, 30, 45 and 60 minutes. The temperature of 420^0C was considered appropriate to avoid formation of either pearlite or martensite. These were to study the effect of the process parameter on both the transformation process, the microstructural evolution and mechanical properties of the materials.

Determination of the Mechanical Properties

The samples were subjected to tests to determine tensile strength, percentage elongation, hardness and impact energy in order to characterize their mechanical properties. The samples were prepared and tests carried out as contained in [10, 4]**.**

The tensile strength and percentage elongation were calculated using the formulae:

$$\text{Tensile Strength} = \frac{\text{Maximum load (N)}}{\text{Original cross-sectional area (mm}^2\text{)}} \quad (1)$$

$$\text{Percentage elongation} = \frac{\text{Elongation at fracture } (L_f - L_0)}{\text{Original guage length } L_0} \quad (2)$$

Where L_f is length at fracture and L_o is original length.

The Brinell hardness number was computed as the value of the load divided by the surface area of the indentation. which is mathematically represented as;

$$\text{Hardness } H_B = \frac{2P}{\pi D[D - \sqrt{D^2 - d^2}]} \quad (3)$$

Where P is the applied load (kg), D is the diameter of the indenter and d is the diameter of the indentation (mm).

Metallographic Investigation

Standard test techniques were used to conduct the microstructural examination of the specimens using metallurgical microscope. The samples were ground roughly with the help of buehlera metaserv 2000 grinding machine using different grits of 60, 120, 400 and 600 grits. Finer and smooth ground surface was achieved by successive grinding from a largest grit to a smallest grit. Polishing was done with diamond paste abrasive of 4 microns' size and further polishing was done with emerald cloth on a rotating polishing wheel. The samples were etched using 3% nital solution containing 3% nitric acid and 97% ethanol for 16

seconds. The microstructure of the samples was thereafter observed usingAP2000MTI metallurgical microscope for microstructural characterization. The images of the micrographs were captured at the magnification of X100.

RESULTS

The chemical composition of the two grades of steel are shown in Table 1 while the mechanical properties for both non-heat treated and the austempered samples for grade A and B steel are presented in Tables 2 and 3 respectively. Figures 3.2A – 3.6A and 3.2B – 3.6B show the various micrographs of the exhibited microstructures in the steel samples for the varied heat treatment conditions

Table 1: Chemical composition of the two grades of steel

Grade of Steel	**Comp. (%)**								
	C	**Mn**	**Si**	**S**	**P**	**Mg**	**Ni**	**Cr**	**Fe**
Grade A	0.56	0.96	0.26	0.05	0.33	-	-	-	Bal
Grade B	0.76	1.11	0.33	0.03	0.05	-	-	0.002	Bal

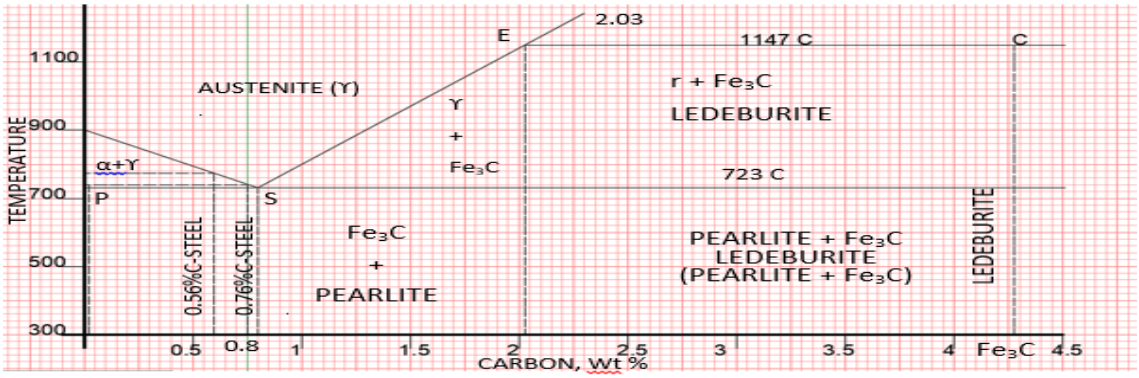

Figure 1: Iron-cementite phase diagram of the steel

Table 2: Mechanical Properties of Grade A Steel

Austenitizing Temperature (^{0}c)	Holding Time (min)	Tensile strength (Mpa)	Hardness (Hv)	Impact Strength (J)	%E
Non heated Sample A		930.2	133.5	10.6	40.3
800	5	1417.11	170.11	32.11	65.21
	15	1351.63	155.71	32.82	81,15
	30	1344.61	130.06	34.32	92.37
	45	1340.30	127.71	32.95	96.25
	60	1301.73	126.35	34.89	97.11
840	5	1356.45	190.51	23.98	56.16
	15	1245.15	178.73	39.62	60.21
	30	1214.06	174.63	42.39	63.15
	45	1188.69	170.41	40.36	66.45

	60	1136.20	160.56	37.27	70.32
900	5	1158.5	205.16	21.13	41.91
	15	1122.28	189.36	24.21	46.36
	30	1116.51	183.57	20.38	47.51
	45	1113.36	178.61	31.96	52.20
	60	1086.91	172.09	45.23	55.51
960	5	946.10	250.98	16.18	29.50
	15	909.60	201.81	17.21	33.21
	30	900.12	180.43	20.21	35.60
	45	894.65	170.99	14.41	38.63
	60	863.51	140.63	21.23	42.10

Table 3: Mechanical Properties of Grade B Steel

Austenitizing Temperature (^{0}C)	Holding time (min)	Tensile strength (Mpa)	Hardness (Hv)	Impact Energy (J)	%E
Non heated Sample B		930.6	132.6	10.6	40.5
800	5	1431.79	201.01	25.63	48.57
	15	1428.16	149.21	26.63	48.57
	30	1423.35	145.22	28.72	48.95
	45	1416.16	140.52	25.54	49.28
	60	1407.20	140.38	27.4	50.78
840	5	1355.07	206.18	17.6	47.21
	15	1330.79	175.16	23.99	47.23
	30	1290.96	155.35	36.42	47.77
	45	1270.98	171.97	34.48	48.54
	60	1229.58	165.29	31.92	48.54
900	5	1169.98	210.0	14.16	44.77
	15	1163.16	195.23	19.17	44.92
	30	1152.74	178.16	22.15	45.94
	45	1135.16	176.06	32.16	47.21
	60	1130.79	175.06	25.54	47.36
960	5	1083.20	260.51	12.06	43.65
	15	1078.71	238.12	12.99	43.71
	30	1072.60	203.33	14.85	44.24
	45	1061.27	197.05	18.07	44.71
	60	1056.80	187.01	15.36	44.77

Figure 3.2A: Sample A Non heat treated

Figure 3.2B: Sample B Non heat treated

Figure 3.3A1: Sample A. at 800^{0}C (5mins)

Figure 3.3A2: Sample A. at 800^{0}C (16mins)

Figure 3.3B1: Sample B. at 800^{0}C (5mins)

Figure 3.3A2: Sample B. at 800^{0}C (16mins)

Figure 3.4A1: Sample A. at 840^{0}C (5mins)

Figure 3.4A2: Sample A. at 840^{0}C (16mins)

Figure 3.4B1: Sample B. at 840^{0}C (5mins)

Figure 3.4B2: Sample B. at 840^{0}C (16mins)

Figure 3.5A1: Sample A. at 900^{0}C (5mins)

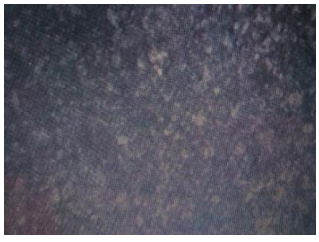

Figure 3.5A2: Sample A. at 900^{0}C (16mins)

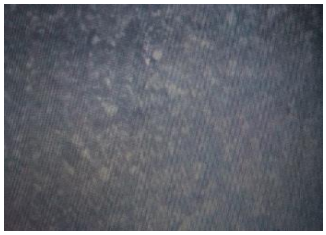

Figure 3.5B1: Sample B. at 900^{0}C (5mins)

Figure 3.5B2: Sample B. at 900^{0}C (16mins)

Figure 3.6A1: Sample A. at 960^0C (5mins)

Figure 3.6A2: Sample A. at 960^0C (16mins)

Figure 3.6B1: Sample B. at 960^0C (5mins)

Figure 3.6B2: Sample B. at 960^0C (16mins)

From Table 1, it is seen that the percentage composition of carbon in samples A and B are 0.56 and 0.76 respectively. Hence, it is evident that both samples of steel are hypoeutectoid steel. [4].
It was observed from the micrographs shown in Figures 3.3 – 3.6, that the structural makeup of the grades of the austempered steel studied were made of bainite, martensite and retained austenite. The presence of these respective phases were indicated in light brown, dark and small darkened block areas in the micrographs.[17, 11, 7].

Effect of the Chemical Composition on the Phase Transformation Process, Microstructure Evolution and the Mechanical Properties of the Steel Samples

Based on the percentage composition of carbon in the two samples of steel, it was observed from the phase diagram presented in figure 1 that austenite formation commenced at the temperatures of 743^0C and 790^0C for sample A and B respectively. This shows that the selection of the austenitizing temperature within the range of 800^0C - 960^0C was appropriate for good dissolution of austentite.

The effect of the percentage composition of carbon on the mechanical properties of the grades of steel was evident. Sample B steel has higher percentage of carbon than that of grade A as shown in Table 1. It has been reported that the higher the carbon content in steel, the more the strength with attendant brittleness. [4]. This postulation correlates with the results obtained in the mechanical property tests. The heat treated samples of B was found to possess more tensile strength values ranging from 1056.8 – 1431.79MPa while in sample A, the values range from 946.1 – 1417.11MPa.

The same reason accounted for the increase in hardness values observed in samples B. It was also noted the higher percentage composition of manganese of 1.11% in sample B must have contributed to increase hardness values as compared in Tables 2 and 3. Manganese has been reported as a carbide-forming element which segregates at the grain boundaries. [11, 18]. It has also been reported that bainitic transformation from austenite proceeds in two stages which eventually leads to the formation of bainite and carbide. [11]. The resultant reactions are shown in the chemical equations represented in equation 1 and 2.

Hence, higher percentage composition of manganese in grade B sample must have enhanced the formation of more amount of carbide observed in the microstructure and illustrated in the chemical equations 1 and 2 which show the transformation of high carbon austentite to bainite and carbide. Carbides are hard and brittle. It can introduce crack which may reduce toughness, and enhance failure of the material. [13].

Therefore, the lower impact strength of sample B confirms the fact that the toughness of the material reduced compared to sample A. In the same way, an inverse trend was observed in elongation where sample B had less elongation values especially at higher temperatures. This shows a strong correlation between the chemical composition and the mechanical properties.

$\gamma \rightarrow \gamma_{hc} + \alpha_b$ (4)

$\gamma_{hc} \rightarrow \alpha_b +$ carbides (5) [11]

The austenite transformation rate was observed to have been influenced by the composition of carbon in the alloys. The samples with higher percentage composition of carbon were found to have less quantity of untransformed austenite with the untransformed austenites shown in dark block colors in the micrograph presented in Figure 3.3 - 3.6. It therefore implies that transformation rate of austenite is more in sample B which is attributed to more nucleation rate. It has been reported that the transformation process is dependent on the nucleation rate due to more nucleation sites [4]**.** This is strongly believed to have decreased the inter phase spacing more in sample B. Such situation could assist carbon atoms to diffuse through a shorter distance and ensure that low carbon regions are enriched.

Effect of the Heat Treatment Process Parameters on the Microstructural Evolution and the Mechanical Properties

The results obtained from the mechanical properties test shows that both austentizing temperature and austempering holding time have significant effect on the properties of heat treated steel. From the micrographs obtained for the microstructural characterization, it was found that the austentizing temperature had significant effect on the grain size. The samples austentized at a lower austentizing temperature yielded finer grain size. It has been reported that more coarse grain size promotes quenching crack and distortion while finer one provides higher strength and toughness [12, 14, 15]. It was therefore confirmed that the higher values of tensile strength and impact strength observed with lower austentizing temperature were traceable to the size of the grains. Furthermore, the effect of austentizing temperature was noted in the hardness of the material. Increase in the austentizing temperature leads to increased in the hardness value. At higher austentizing temperature, more amounts of carbides could be dissolved. This increase in the carbon content in the martensite which in turn gives rise to lattice strain was as a result of super saturation of carbon [12]. The outcome of these processes contributed to increase in the hardness value as the austentizing temperature increased.

The effect of the austempering holding time in the bainitic reaction process was observed in the micrographs and confirmed in the result for the mechanical property test. Comparison of the micrographs of samples soaked for 5 minutes and those soaked for 15 minutes shows that longer austempering holding time yielded higher quantity of bainite due to progressive increase in the bainitic transformation. This is evident in the microstructure which revealed more of the light brown colour of the bainites in the samples held for longer time. It was also noted that the increase in the amount of bainite with time in the microstructure depreciated the level of martensite. Martensite is a hardened micro constituent of the steel structure which enhances hardness property. Therefore, the observed decrease of martensite showed a drastic decrease in the hardness value of the steel material with progressive increase in holding time at each austentizing temperature.

The highest value of hardness which was recorded for the shortest holding time of 5 minutes is traced to the low level of bainitic transformation at such earlier time of austempering transformation. This caused super saturation of carbon in the retained austentite which induced high lattice strain in the martensite formed after cooling [17, 18]. When the holding time increased to 60 minutes, it was observed from the microstructure that more of the austenites were transformed to bainite which left less quantity of the austentite for martensitic transformation. The transformation which led to the formation of more bainite and carbide precipitation as presented in the equation 4 is one of the major factors that reduced the lattice strain and decreased the hardness as the holding time progressed. It was observed that for both samples of steel, in all the austentizing temperature, hardness values tend to stabilize after 30 minutes holding time. Within the holding time range of 45 - 60 minutes, the hardness value slightly increased unlike in the previous early times. Such deviation in the hardness behavior is attributed to the completion of bainitic transformation. At such stage, there could be an increase in the conversion of retained austentite to martensite [17, 18]. In contrast, elongation showed a reverse trend with time hence it was noted that the soaking time showed a direct relationship with elongation and an inverse relationship with hardness for both materials.

From the results obtained from the impact strength test, it was noted that impact strength is dependent on the nature of the alloy, the phases developed in the microstructures and the heat treatment process parameters. For sample A, the increase in austentizing temperature from 800^0C to 960^0C did not show a regular increase or decrease in the values of impact strength. The values obtained decreased and increased in different austentizing temperature points. However, it was observed that the values obtained depend more on the holding time. For instance, at 800^0C, the value of impact strength increased from 32.11J to 34.89J when the austempering holding time increased from 5 to 60 minutes. With further elevation in austentizing temperature to 840^0C, the value increased to 42.39J between 5 to 30 minutes. However, the value finally decreased from 42.39J to 37.27J between 30 – 60 minutes holding time. Between 900^0C - 960^0C, the impact strength behavior showed an irregular increase and decrease with time with maximum and minimum values of 45.23J and 14.41J at 900^0C (60minutes) and 960^0C (45minutes) respectively. In sample B, the same trend was observed in impact strength behavior. However, maximum increase in the value was noted at intermediate austempering holding time.

The increase in the impact strength is traceable to large amount of bainite while the decrease could be attributed to large amount of solute hardened retained austentite and martensite in the microstructure as reported by [17, 18]. The intermittent rise and fall in the values observed at different temperature and time could be explained by the changes in lattice strains caused by phase changes due to variation in the process conditions. Notwithstanding, the reliability of the heat treatment equipment could be queried for such irregularities.

Finally, it was found that for both samples, higher tensile strength value was associated with shorter soaking time range and is due to higher nucleation rate at that period which yielded finer grain structure. With further increase in the soaking time, nucleation ceased, as grain growth was slightly initiated which grew with time. Excessive grain growth which yields coarse grain structure has been reported to have the tendency to impair the strength of the material. [12, 14, 15]. This accounted for improved strength and

other related properties at the earlier soaking time when compared to the ones obtained at a longer holding time.

Therefore, this shows that the phase transformation which yielded the microstructures obtained in respect to the heat treatment process parameter correlates with the mechanical properties of the developed steel samples as earlier stated by [12]. Notwithstanding, it is evident from the result that the mechanical properties is also a function of the chemical composition of the steel.

CONCLUSIONS

From the findings made in this study, the following conclusions were established;
The chemical compositions, heat treatment conditions and the phase changes have a strong correlation with the microstructures and the mechanical properties of austempered steel. The interrelationship between these process parameters and the properties is such that one of the factor has effect on the other factor.

For hypoeutectoid steel, the sample with higher composition of elements such as carbon and manganese responds better to austempering heat treatment process to yield better mechanical properties.

For the two samples of steel, the most promising microstructures with respect to excellent strength-ductility property are those obtained at the ausenitizing temperature range of 800 -840^{0}C and soaked within the time range of 5 – 30 minutes.

The mechanical properties and microstructural characterization confirm that decreasing austenitizing temperature yields finer grain structures with increased tensile strength and elongation with decrease in hardness. However, with little effect on the impact strength at any given austenitizing temperature, shorter austempering holding time yielded optimum properties in tensile strength and elongation while higher hardness values were associated with longer holding time.

The microstructure obtained in the austempered steel austenitized within the range of 800 - 840^{0}C using austempering time range of 5 – 30 minutes exhibited potential for structural and load bearing applications while those austenitized within the range of 900 - 960^{0}C using austempering time range of 5 – 15 minutes are candidate material for wear resistant application.

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