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Rapid Cyclic Heating of Mild Steel and its Effects on Microstructure and Mechanical properties

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Abstract. The effect of subjecting mild steel to several cycles of rapid heat treatment on its mechanical properties and microstructure was investigated. Mild steel of 0.213 wt % carbon was subjected to transformation heat treatment from austenite to pearlite and quenched in running water. Rapid heating was achieved by preheating an electric muffle furnace to 840 °C before charging the samples into it. Each cyclic heat treatment was for a period of 200 seconds held at 840 °C and cooled to 700 °C which was repeated four times. The effects of cycle numbers were evaluated by testing for impact, hardness, ultimate tensile strength and microstructural properties. The results showed that after 4th cycle of rapid heating the samples had impact energy 64.6 J, Brinell hardness number 563 and ultimate tensile strength 1257.78 N/mm². The samples after one cycle had ultimate tensile strength 1027.45 N/mm², % elongation 10.396% and impact energy 286.174 N.m before failure. Through cyclic heating, grain refinement was achieved by the fast simultaneous nucleation at the grain boundaries and the fast martensite to austenite transformation due to the fast heating rate which prevented austenite grain growth. Mechanical properties of the studied steel sample were improved with the rapid heat treatment cycles given.

Keywords: Cyclic heating, rapid heat treatment, microstructure, quenching.

1. Introduction

Steel is one of the widely used metals and exist as low, medium and high carbon steel. It can be strengthened through additional heat treatment processes Jiang and Yazheng [1] which make it to be adopted for various applications. Low carbon steels (0.002 - 0.25% C) account for a large proportion of the total output of steel due to its relatively low price and adaptable properties similar to that of iron therefore making its application versatile for many engineering applications as observed by Adamczy and Grajcar [2]. Low carbon steels are used for different applications as in automobiles, furniture, refrigerators and tinplate roofing. In between 0.15 and 0.25% C content is used for structural purposes such as beams, channels and angles for construction.

The choice of heat treatment adopted for strengthening steel is very important as faulty heat treatment process may incur high cost of operations and untimely failure of materials while in service which may have effect on human safety (Senthilkumar and Ajiboye [3]. Muszka *et al.*; [4] opined that the specific mechanical properties of metals are the basic requirements for its application. These properties can be obtained from different strengthening mechanisms which act together during deformation process. Metal strengthening procedure depends on its chemical composition, deformation history and resulting microstructure.

There are various methods of strengthening that include solid solution strengthening, dispersion strengthening, grain refinement, dislocation strengthening and textural strengthening as submitted by Lv, *et al.*; [5]. The rate of metal cooling from the austenitic temperature range as in Aweda and Adeyemi [6] has great effect on structure and properties of the material. They observed that fast cooling rates result in phase transformation at lower temperatures thus yielding finer grain structure than slow cooling.



Fast cooling rate transforms steel from its austenitic phase to a new martensitic phase Akinlabi *et al.*; [7]. During quenching, the cooling rate is controlled to avoid soft pearlite or bainite formation. Therefore, the method adopted for quenching depends on the grade of steel, section thickness, distortion allowed and the properties imparted in the opinion of Roney and Loker [8].

Grain refinement can simultaneously enhance strength and toughness of materials which leads to the development of high performance materials, Lv *et al.*; [5]. Calcagnotto *et al.*; [9] achieved grain refinement by subjecting dual-phase steel to large strain warm deformation and subsequent inter-critical annealing that led to improved strength and ductility of the product. In addition to other procedures of steel processing, Jiang and Yazheng [1] reported that cyclic rapid heat treatment can be adopted to improve its properties.

The possibility of refining the austenite grains through shortening the austenitizing cycle by subjecting steel to rapid heat treatment involving heating steel at a fast heating rate to its austenitic temperature and quenching immediately without soaking was attempted by Grange [10]. In his submission, he argued that soaking the steel at the austenitizing temperature was not really necessary, adding that the process of very short or rapid austenitizing treatment has the advantages of saving time, cost, minimizing the oxidation and decarburization of steel and minimizing austenite grain growth thereby leading to a finer grain heat treated product. As at the time of his work, he opined that two or more cycles of rapid heat treatment would improve the ductility of mild steel. Improving on Grange [10] submission, Koscielna and Wojciech [11] and Zheng *et al.*; [12], applying cyclic heating process to Ti-48Al-2Cr-2Nb alloy discovered that the average grain diameter reached its minimum value of 149 μm after 5 cycles while further increase in the number of cycles had no effect on the grain size.

With rapid heat treatment, a hard surface with improved fatigue and wear resistance is achieved Boyce *et al.*; [13]. Thermal cycling, as observed by Saha *et al.*; [14] produced fine microstructure with a good combination of strength and ductility. This was in agreement with Smoljan [15] who concluded that repeated heating is required to have improved effects of structure refining than single heating. He further stated that the number of cycles affects the obtained results of heat treatment and suggested five cycles. Above this number, micro-cracks may occur that can affect toughness as well as yield strength, Lin *et al.*; [16].

With insufficient cycle number, structure refining may not be obtained while at too many cycles, defects may occur due to micro-deformation processes which may not be economical. Number of cycles of rapid heat treatment depends on the material and ranged between three to maximum of six cycles depending on the material. This study therefore, examines the effects of cyclic rapid heat treatment behaviour of mild steel on its mechanical and microstructural properties.

2. Experimental procedures

Commercial mild steel was used for the experimental investigation. The analytical chemical composition of the as-received steel is as presented in Table 1 using optical emission spectroscopy. The steel samples dimensions 10x10x55 mm were subjected to isothermal heat treatment at 840°C for 200 seconds and then cooled to room temperature. The as-transformed steel were subjected to different number of rapid heat treatment cycles thus; 1-cycle, 2-cycle, 3-cycle and 4-cycle as shown in fig 1. For each rapid cycle heat treatment, the specimens were inserted into an electric resistance furnace at 840°C (above Ac₃ temperature 754°C) and holding for 200 s. This was followed by quenching to 700°C with a cooling time of 60 s. The specimens were thereafter air-cooled to room temperature, which ends 1-cycle heat treatment. Each cycle consisted of inserting the specimen in an electric furnace at 840°C and holding for 200 seconds followed by forced cooling to 700°C. This procedure was repeated for the samples subjected to more than 1-cycle rapid heat treatment. Samples with 2-cycle heat treatment were repeated once, 3-cycle repeated twice and 4-cycle heat treatment repeated thrice, see figure 1.

Table 1: Compositional analysis of tested mild steel (wt%)

Element	Fe	Cr	C	Si	Mn	S	P	Ni	Cu	Nb	Al	B	Mo	Ti
% wt	97.75	.138	.213	.252	.779	.033	.028	.134	.336	.014	.292	.002	.020	.009

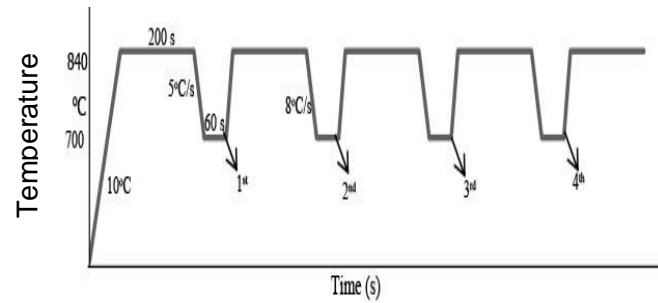


Figure 1 Cyclic heat treatment plots

The hardness values of the specimens were measured on the Brinell hardness testing machine performed in accordance with ASTM E10-15 for metallic materials. A 750 kgf was maintained for 15 seconds on the sample and the indenter was then removed leaving a round indent on the sample. The diameter of the indent was determined optically by measuring two diagonals of the round indent with a portable microscope and the values of Brinell hardness were calculated following equation 1.

$$\text{BHN} = \frac{2F}{\pi D(D - \sqrt{D^2 - d^2})} \quad (1)$$

Where,

BHN = Brinell Hardness Number

F = applied load, kg

D = the diameter of the spherical indenter, mm

d = diameter of the resulting indenter impressions, mm

Impact test was conducted on the standard Charpy V-notch impact tester following ASTM A370, ISO148 and EN 10045-1 procedures. The standard specimens' size of 10 mm by 10 mm by 55 mm and 45° angular groove at the middle with a depth of 2 mm was cut and tested for impact. Following ASTM-E8 procedure for tensile test, a UTM Tensometric S300CT of maximum load of 300 kN at a crosshead speed of 0.03 mm/s. The specimen was loaded gradually until it failed and the value of stress with corresponding strain diagram was plotted electronically for all the tests. From the data obtained the % elongation, yield strength, ultimate tensile strength and Young modulus values were determined. The microstructural observations of the as-transformed and cyclic heat treated specimens were conducted on optical microscope. Silicon carbide papers of different grades placed on the grinding machine were used in the order of 220, 320, 400 and 600, from coarse grade to fine grade. The polishing was performed on a universal polishing machine with a polishing cloth (selvet cloth) and solution of one micron of silicon carbide solution. This was followed by the final polishing stage with selvt cloth swamped with solution of 0.5 μm silicon carbide until a mirror-like surface was attained. The samples, washed and dried were then etched with 4 % NITAL and again washed and dried.

3. Results and discussion

3.1 Hardness values

The effect of Brinell hardness values on cycle numbers is shown on figure 2. The Brinell Hardness Number (BHN) values increase with increase in the number of rapid heating cycles from 271 for as-transformed (control) to 323 after 2-cycles of heat treatment. The increase continued until the value of 563 was reached after four cycles of rapid heating. This increase was probably due to the material's resistance to plastic deformation as the number of grain boundaries increased during quenching. This

trend agrees with Xie *et al*, [17] results where the hardness of mild steel improved through rapid cyclic heating and quenching. This is because subjecting steel to fast heating rate leads to the formation of fine austenite grains which on quenching produces coarse martensite. The coarse martensite grains are refined into finer martensite grains with more grain boundaries upon cyclic rapid heating.

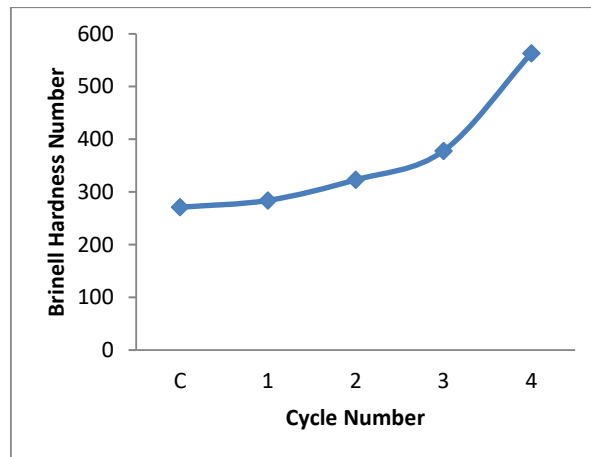


Figure 2 Effect of Brinell hardness on cycle numbers

3.2 Charpy V-Notch Impact Test

Figure 3 is the Charpy V-Notch impact test results with cycle numbers which shows increase in impact energy from 46.8 J for the as-transformed (control) sample to 51.2 after the first cycle heat treatment. There is further increase to 64.6 J with the accomplishment of the 4th cycle of rapid heating. This steady increase indicates that the resilience of the material increases with subsequent increase in cycle number of rapid heat treatment. This is an indication that mild steel possess ability to withstand shock loading with increase in cycle number as its martensitic structure is refined.

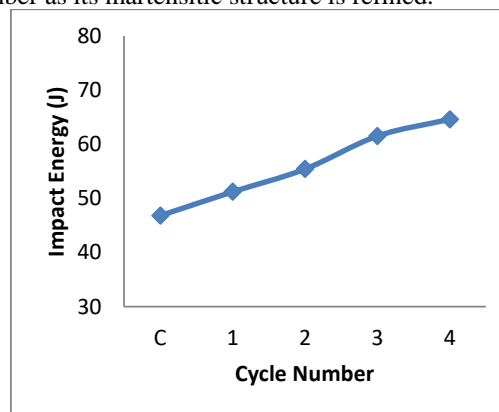


Figure 3 Impact strength of sample with cycle number

The percentage increase in impact strength from the control sample to the first cycle heating is 9.4%, while the increase in impact strength between the first cycle and the second cycle is 8.2%. The percentage increase impact strength between the second and third cycle samples is 11.01% while the difference in the energy between the third and fourth cycle samples was 5.04%. From these results, further increase in cycle numbers of rapid heat treatment may lead to minimal increase in impact strength. After a substantial increase in the 3rd cycle, subsequent increase is marginal with increase in cycle number.

3.3 Stress-strain curve

The stress-strain curves from the tensile tests of the control (as-transformed) and cycle heat treated samples are as obtained in figure 4. The control sample failed within the elastic limit, suggesting brittle failure but with a high ultimate strength of 1247.78 N/mm². This indicates that conventionally, diffusional heating and quenching produces high ultimate strength. The first cycle yielded at lower ultimate strength value of 1027.45 N/mm².

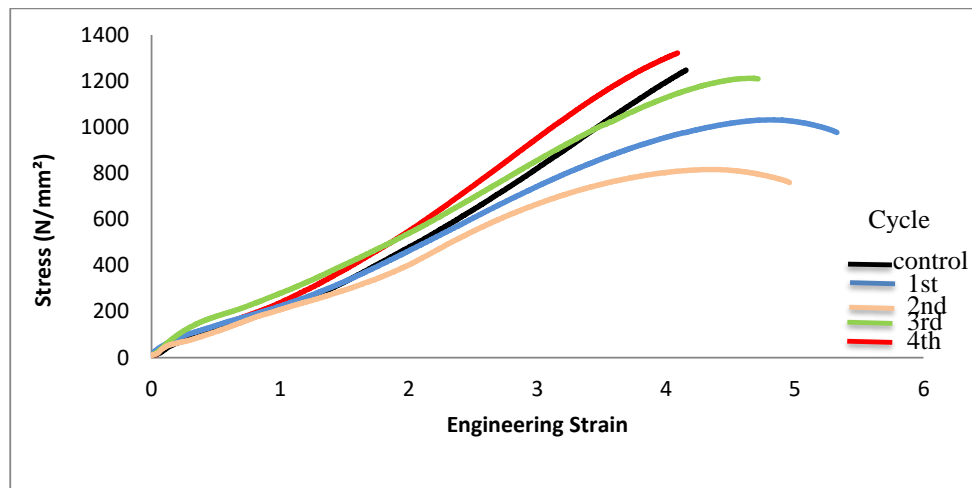


Figure 4 Strain-strain curves

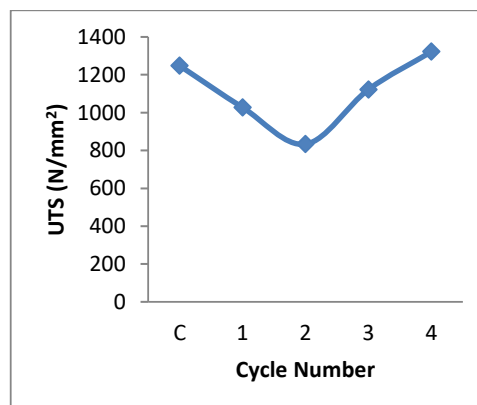


Figure 5 UTS versus cycle numbers

The yield strength initially dropped for the 1st and 2nd cycles only to rise with the 3rd and 4th cycles of heat treatments. The yield strength for the control was 1247.45 N/mm² while this value increased to 1321.78 N/mm² after the 4th cycle of heat treatment as in figures 4 and 5. After the second cycle, the material experienced plastic deformation with recorded lower ultimate strength value of 833.375 N/mm². This shows that the second cycle is more ductile than the first cycle sample. The third cycle also underwent plastic deformation but with a higher ultimate strength than the first and second cycle recording 1122.24 N/mm². These results show a reversal of trend as there is a loss in ductility. The fourth cycle sample failed in a brittle manner as it did not undergo plastic deformation. It has the highest ultimate strength of 1321.78 N/mm² before failure occurred. Smoljan [14] explained that during rapid cyclic heat treatment, the cycle number should not be too many hence it will lead to loss of ductility in relation with strength. This explains the behaviour resulting from the third and fourth cycle samples.

3.4 Effect of cyclic heat treatment on yield strength

The control sample with 1247.75 N/mm² and 4th cycle sample with a value of 1321.78 N/mm² becomes the highest yield strength. This shows that the material's resistance to the movement of dislocation was due to their grain boundaries as displaced in figure 5. The yield strength is lower in the 1st cycle been 640 N/mm². This occurrence was due to the fact that ductility was beginning to set in as ductile materials have lower yield strength compared to brittle materials. This value reduced further in 2nd cycle to 480 N/mm² before witnessing a rise in the 3rd cycle to 850 N/mm². This is an indication that the 2nd cycle sample is the most ductile while the 3rd cycle experience loss in ductility. The feasible yield point can be attributed to the dislocation pie up on the ferrite grain boundaries and the dislocation activation in the ferrite grains, Lv *et al.*; [5]. The stress-strain curves from the tensile tests of the as-transformed and cycle heat treated samples are as shown in figure 4.

3.5 Effect of cyclic heat treatment on ultimate tensile strength

The values of the ultimate tensile strength for the control and rapid cycle heat treated samples are as presented in figure 5. UTS increases and followed thereafter by a decrease. The initial increase was due to the finer microstructure (finer pearlite and ferrite) originated from non-equilibrium forced cooling in each treatment. These values are however the highest values for the ultimate tensile strength which is a measure of a material's ability to withstand deformation due to tension. The values for the ultimate tensile strength for the 1st, 2nd and 3rd cycles are 1027.45 N/mm², 833.375 N/mm² and 1122.24 N/mm² respectively. The 1st cycle sample has high UTS, while the lowest was recorded at the 2nd cycle. The results show that as the material undergoes more heat cycles, the particles get finer, the materials become more ductile and as a result the ultimate tensile strength decreases in value for 1st and 2nd cycles and thereafter, increasing to 1405 N/mm² at the 4th cycle.

3.6 Effect of cyclic heat treatment on % elongation

Figures 6 and 7 show the 2nd cycle has a high ductility as it records high % elongation. The yield point is 8.556 with a percentage elongation of 9.697% and Young modulus value of 15356.3 N/mm². With increase in cycle number, the % elongation of the material reduces from 10.4 % for 1st cycle to 8.18% after the 4th cycle while the as-transformed was 8.32%. The 1st cycle sample and 3rd cycle sample also showed ductility with percentage elongations of 10.396% and 8.912% and corresponding Young modulus values of 16398.5 N/mm² and 20616 N/mm² respectively. The control sample and 4th cycle sample showed the lowest % elongation which also translates to lowest ductility which explains why they have the highest ultimate tensile strength.

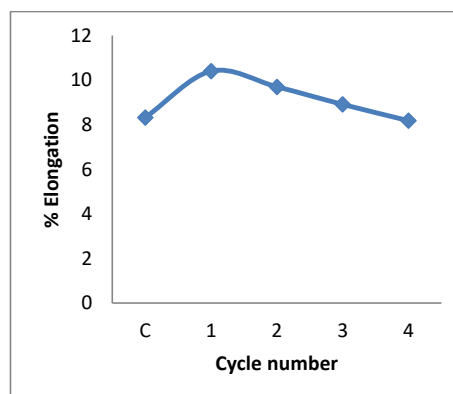


Figure 6 Percentage elongations of samples

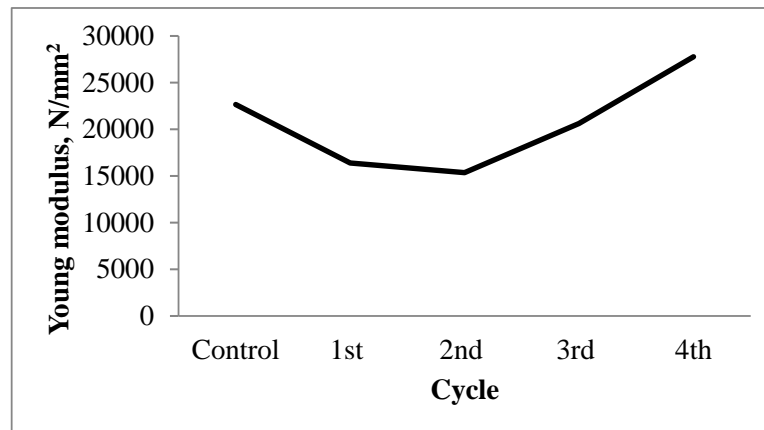


Figure 7 Young modulus values of samples

3.7 Effect of cyclic heat treatment on toughness

The energy absorbed before failure with cycle number on figure 8 showed that the 1st cycle absorbed 296.174 N.m while the control sample had 230.981 N.m before failure. Subsequent cycle heat treatment numbers produced lower energy compared to the 1st cycle. This makes the 1st cycle sample the toughest implying that one cycle of rapid heat treatment is enough to produce toughness in mild steel.

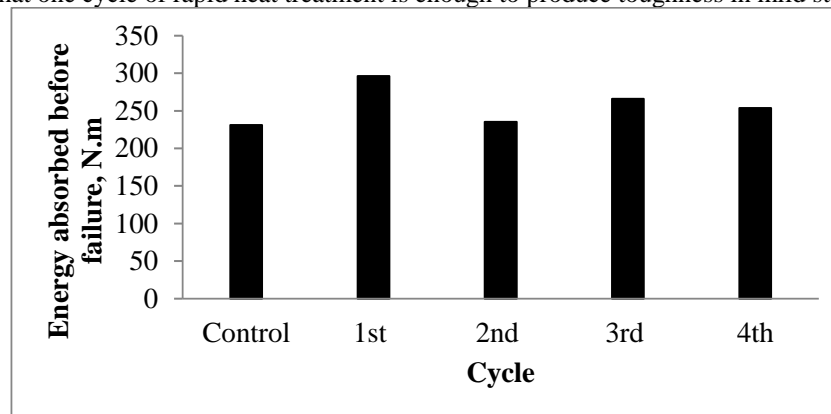


Figure 8 Energy absorbed before failure with cycle number

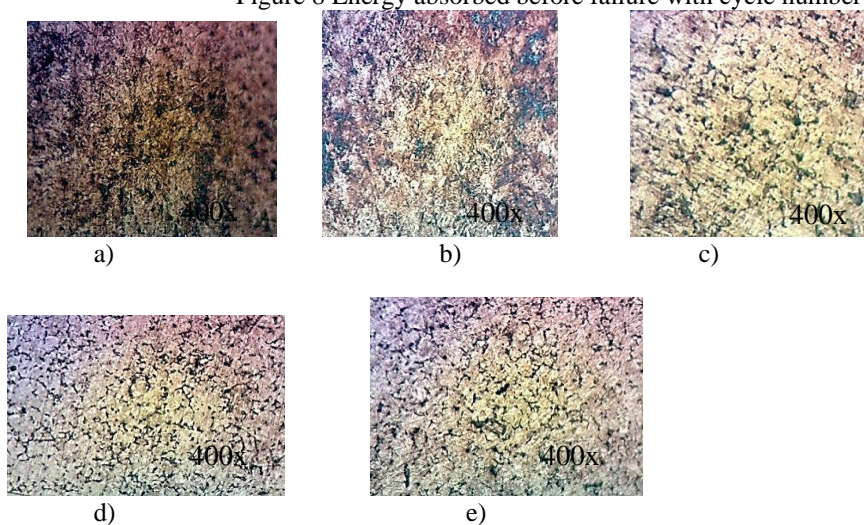


Figure 9 Optical micrographs of amples

a) Control Sample b) 1st Cycle sample c) 2nd Cycle sample d) 3rd Cycle Sample e) 4th Cycle sample

Table 2 Summary of Microstructure Properties

Cycle	Count	Total Area (μm^2)	Grain Size (μm)	Circularity	solidity	Feret (μm)
Control	2499	1153849	1.07	0.801	0.856	0.032
1 st	2877	1152582	1.00	0.80	0.853	0.30
2 nd	970	249739	0.80	0.769	0.843	0.38
3 rd	2633	663536	0.79	0.793	0.854	0.036
4 th	2980	859164	0.85	0.799	0.856	0.033

3.8 Effect of cyclic heat treatment on microstructure

As the specimens were held above Ac3 temperature, ferrite-to-austenite transformation occurs and the cementite lamellae begin to dissolve into austenite. The as-transformed indicates isothermally transformed pearlitic characteristics. For the specimen that went through one cycle rapid heat treatment, the cementite is partially dissolved into pearlite while there is further dissolution after 2-cycles of heat treatment. During holding after heating, the cementite dissolves and upon cooling the dissolution is terminated. The microstructure obtained by cyclic rapid heating and cooling of steel as presented in figures 9(a-e) show martensite in solution of ferrite Roney and Loker [8]. The carbon devoid of austenite regions converts into ferrite upon forced cooling as is noted from figures 9 b and c with rapid cyclic heating. With as-transformed figure 9a, cementite phase is present initially within pearlite. With increasing number of cyclic heating figures 9 c and d, the amount of pearlite decreases while the amount of cementite increases. After the 4th cycle figure 9e, the microstructure mainly contains cementite in ferrite with only trace amount of pearlite. In the process of austenization, the ferrite to austenite transformation occurs very fast, whereas the cementite dissolution in austenite is a slow process in accordance with Saha *et al.*; [14]. This is because the fast cooling rate did not allow the carbon particles to diffuse evenly into the matrix of the iron. This results in the jamming of the carbon lattice of the austenitic atomic arrangement. Martensite, an aggregate of iron and cementite has grains that are very coarse hence the mottled contrast seen in figure 9a is due to the high density of dislocation. Rapid heating allows some of the martensite particles to transform directly into austenite without passing through the intermediate phases. The carbon particles however were given more room to diffuse out and realign thereby leading to more evenly distributed microstructure on quenching. The process results in producing finer grains due to nucleation of grains and formation of grain boundaries as observed in figure 9e.

This trend is evident in Table 2 as the control sample possessed the largest grain size of 1.07 μm (microns). There was a reduction in the grain size after the 1st cycle to 1 micron which further reduced to 0.79 microns after the 3rd cycle. This is because more grains are beginning to form due to nucleation as the material is heated rapidly and quenched. The circularity of the grains which indicates the shape of grains, reduced from 0.801 for the control sample to 0.769 after the 2nd cycle before experiencing an increase of 0.793 after the 3rd cycle show grain growth occurring. The solidity of the grains (thickness) also followed similar pattern where there was initial decrease up to the 2nd cycle followed by an increase thereafter. The distance between grain boundaries (Feret) also varied from control cycle through the four cycles. The largest value was recorded in the 2nd cycle of rapid heat treatment with a value of 0.038 μm .

The resultant microstructures obtained and the mechanical properties obtained show that rapid heating leads to a refinement in the grain size and improvement in mechanical properties such as ductility, toughness, ultimate tensile strength, impact and hardness of mild steel. The improvement in mechanical properties of cycle rapid heat treated material was achieved by the microstructural refinement due to the phase interface nucleation as observed by Peng *et al.*; [18]. Rapid heating prevents the growth of austenite grain size which favours nucleation therefore, on quenching, the martensite microstructure is refined. Nucleation rate is accelerated by refining the austenite grain size as the density of nucleation sites increases inversely with the austenite grain size. After 4 cycles of heat treatment, hardness

marginally decreases partly due to elimination of pearlite and generation of more cementite spheroids in the microstructure.

4. Conclusion

- i. The sample with the 4th cycle of heat treatment possessed high Brinell hardness value and UTS and would find application where high strength is required.
- ii. To obtain finer grains and improve toughness and ductility, mild steel should be subjected to a minimum of three and maximum of four cycles of rapid heat treatment. Any further cycle of rapid heat treatment will reverse the trend.
- iii. Grain refinement was achieved by the fast simultaneous nucleation at the grain boundaries and the fast martensite to austenite transformation due to the fast heating rate which prevented austenite grain growth.
- iv. For the situation requiring tough material in service, one or two cycles of rapid heating is adequate.
- v. Young modulus initially decreases due to generation of finer structure and thereafter slightly increases with gradual elimination of pearlite in the microstructure.

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