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Effects of Annealing Soaking Time on Carbide Precipitates and Mechanical Properties of As-Cast Thin Wall Ductile Iron

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Abstract: *Thin Wall Ductile Irons (TWDI) are viable material option in the automotive industry due of their good castability, adequate mechanical properties, wear resistance, good machinability and fatigue properties. However due to size reduction and increased cooling rate, carbide precipitates occur in the as-cast microstructure of TWDIs, impacting negatively on mechanical properties, nodularity and nodule count. Heat treatment processes that reduce or eliminate these carbide phases can be adopted to remedy such defects. In this study, 4 mm TWDI samples cast in green sand moulds, showing carbide precipitation in microstructure were subjected to an Annealing heat treatment by austenizing to 920^{o}C and varying soaking times of 5, 10, 15, 20, 25 and 30 minutes, afterwards they were cooled in the furnace to room temperature. Microstructural analysis and mechanical property tests were carried out in all the samples via Optical microscopy (OM) and Scanning electron microscopy (SEM), hardness and tensile tests. Microstructure of heat-treated samples showed significant reduction of carbide precipitates in comparison with their as cast counterpart. It was observed that the soaking time impacted on carbide precipitation reduction. Hardness values reduced from 291.6 Hv in the as cast sample to 247.8 Hv and 243.5 Hv for samples soaked for 25 and 30 minutes respectively, whereas tensile strength reduced progressively from 596 MPa to 544 MPa for as cast to 30 minutes soaking time respectively. Percent elongation increased from 4.3 % elongation for the as cast sample to 8.7 % elongation in the sample with soaking time of 30 minutes at 920^{o}C. This study has shown that carbide precipitation can be significantly reduced by adopting the annealing treatment parameters outlined above.*

Keywords: *Carbide Precipitates, Microstructure, Graphite Nodules, Heat-Treated, Soaking Time, Samples.*

1. INTRODUCTION

Ductile Iron (DI) thin section profiles of thickness $\leqslant$ 5mm are called thin wall ductile irons (TWDI) [1]. In comparison with other lightweight automotive parts like aluminium, TWDIs offer better mechanical properties in terms of high strength to weight ratio. Engineers in automobile part manufacture are often faced with the need for fuel consumption reduction, emission control and manufacturing cost reduction, therefore reduction of vehicle weight becomes imperative in order to save material and energy. Some examples of these automotive parts where TWDIs are currently adopted for lightweight parts for energy saving are control arms, connecting rods, exhaust manifolds and wheel rims [2].

However, TWDIs are susceptible to carbide precipitations due to reduction in size, in the event of this, these castings are rejected and the huge energy, man hour and other resources adopted for their production is wasted. Higher cooling rates favour the formation of eutectic carbides whereas slower cooling favours graphite precipitation. Carbide precipitation in TWDIs is shown in Figure 1, whereas graphite nodules in DIs are shown in Figure 2. The white phases in Figure 1 show M3C type eutectic carbides which were obtained by eutectic reaction L $\rightarrow$ γ + M3C. These carbides are distributed originally in the boundaries of the austenite grains, and seriously impact negatively on DI toughness. Carbides that form during the casting process are referred to as primary carbides, whereas those formed during heat treatment are known as secondary carbides. Secondary carbides are typically of an extremely fine nature and serve to strengthen the matrix of the material [3]. Primary carbides are hard and brittle carbides that forms when there is excess carbon in the iron matrix [4]. It appears as white, needle-like or platelike structures within the microstructure. Cementite can contribute to increased hardness but can also reduce ductility and impact resistance, making it undesirable in most cases [5]. These carbides can be greatly reduced by ensuring the use of mould making materials that enable favorable cooling rate for precipitation of adequate microstructure is adopted. In the study of [6], the thermal properties of the molding sand were modified by addition of rice husk ash (RHA) to silica moulding sand, the resulting molding sand mix showed significant reduction in the thermal conductivity. This impacted positively on microstructure by impeding carbide precipitation. During solidification, the formation of eutectic carbides affects the volume fraction of graphite produced as graphite and carbide compete for carbon contained in liquid iron [7].

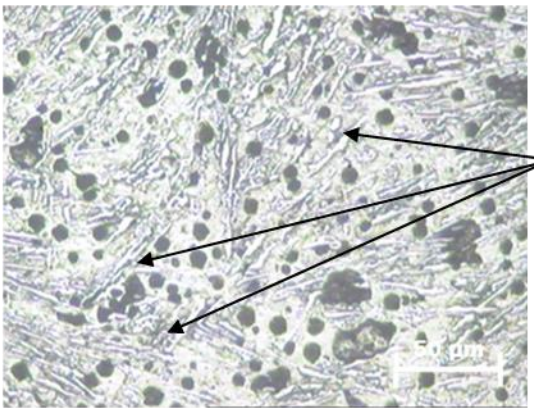

Figure 1: Carbide precipitation in TWDI [8]

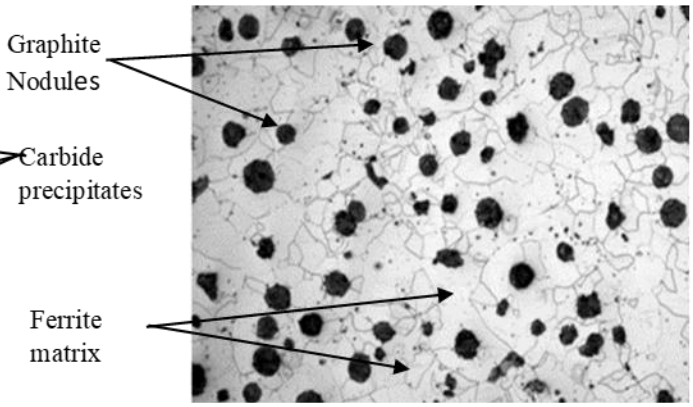

Figure 2: Graphite nodules in DI [9]

Chemical composition of charge materials when casting TWDIs is of great importance during casting as the existence of carbide forming elements and quantity of silicon in the melt significantly influences carbide forming tendency and the degree of graphitization in the sample. In the study of [10], the researchers investigated the impact of carbide-promoting elements: Chromium (Cr), Manganese (Mn), Molybdenum (Mo), Niobium (Nb) and Vanadium (V) on morphology of graphite, pearlite and carbide fractions in high-silicon DI with 3.8 wt.% Si. The conclusion was that the elements analysed negatively impacted on growth of spheroidal graphite and they promoted the formation of carbides and pearlite during eutectic solidification and eutectoid transformation respectively.

Heat treatment which is the controlled process of heating or cooling of a metal to change or modify the existing microstructure and bring about change in its mechanical properties remains an important tool in processing of engineering materials. In [11], the researchers conducted a review on heat treatment, phase evolution and mechanical characterization of cast iron. Their study concluded that the isothermal heat treatment process as a unique method for producing graphite cast iron with exceptional mechanical properties.

Heat treatment processes such as austempering, normalizing and annealing are carried out to improve mechanical properties of DIs, in the austempering process, the specimen was placed in the furnace and heated to the temperature of 860˚C, and then water quenched and cooled. In the normalizing process, the specimen was placed in the furnace and heated to the temperature of 925˚C, then air cooled. In the annealing process, the specimen was also placed in the furnace and heated to the temperature of 900˚C, then furnace cooled to room temperature. Maximum hardness was observed in samples austempered at 860˚C, whereas minimum hardness value was achieved in samples annealed at 900˚C. Tensile strength values were maximum in samples normalized at 925˚C [12].

In [13], the researchers studied the influence of austenitizing process on the mechanical properties of TWDI connecting rod, TWDI connecting rod component was subjected to austempering process. Various holding times of 15, 30, 45 and 60 minutes were adopted using austenitizing temperature of 960˚C and austempering temperature of 350˚C with 35 minutes isothermal time. The process was done in fluidized bed furnace.

In this study TWDI samples that showed large volumes of carbide precipitation in their as cast microstructure were subjected to annealing treatment process using varying soaking times to determine the effect on reduction or possible elimination of these carbide precipitates.

2. METHODOLOGY

2.1 Materials

As cast TWDI samples with carbide precipitates, metallographic consumables, heat treatment furnace, pyrometer, foundry tongs, hand gloves, optical spectrometer, Nikon eclipse ME600 Metallurgical microscope, grinding machine with emery papers, polishing machine and cloth, portable hacksaw, angle grinder all located at the laboratory of Metallurgical and Materials Engineering Department, University of Lagos, Akoka.

2.2 Casting of TWDI Samples

The TWDI samples were cast in Foundry section of Nigerian Machine Tools Limited, Oshogbo, Osun State, Nigeria, the materials for casting were sourced from the Foundry. Green sand moulds prepared from silica sand was adopted as the mould material. Two rectangular wooden patterns of dimensions 4 x 300 x 300 mm were used for preparing the mould. Graphite coke, cast iron sleeve scrap and ferrosilicon alloy which were the charge materials were melted in 500 kg Induction furnace, to the melting temperature of 1450^{o}C, and then superheated to 1500^{o}C to ensure adequate fluidity of the melt for proper filling of the mould cavity. The melt was then poured into a preheated treatment ladle for nodularization via the sandwich process. Ferrosilicon magnesium (FeSiMg) alloy was used for nodularization treatment after melting. Two stage inoculation treatments were done for the melt before pouring into the mould cavity. The mould cavities were two in number located in the drag side of the mould whereas the sprue was located in the cope. Pouring into the mould

cavity was done at melt temperature of 1390°C. The samples were allowed to cool in the mould cavity, after which fettling was done, then spectrometric analysis was carried out to determine the elemental composition of the samples.

2.3 Heat Treatment of Cast TWDI Samples

The samples were machined for microstructural analysis: optical, scanning electron microscopy, X-ray diffraction and mechanical property tests: tensile and hardness. The samples were then placed in the furnace and heated to 920°C, the austenization temperature adopted for austenization process, for the soaking times, six samples representing six soaking times of 5 mins, 10 mins, 15 mins, 20 mins, 25 mins and 30 mins were adopted. The samples were charged into the furnace separately, after the soaking times were achieved, the samples were then furnace cooled to room temperature. This process is called annealing heat treatment.

2.4 Hardness Test

The hardness test of all the samples, both the heat-treated and as cast were evaluated according to specification outlined in ASTM E384 standard for Vickers micro indentation hardness testing. The samples were placed on the anvil of the testing machine and the diamond indenter was positioned on the flat surface of the samples with the load for the required time, and then released, the diagonal lengths of the resulting indentation were measured using the optical system. This was repeated for three times at different locations on each of the samples, then the mean value was recorded.

2.5 Tensile Testing

Tensile tests were carried out according to specifications outlined in ASTM E8 standard for flat samples, using a Universal Instron 3369 Tensometer, located at the laboratory of the Materials and Metallurgical Engineering Department, University of Lagos. The samples were machined to specifications according to ASTM E8 standard as shown in Figure 3. The TWDI samples were mounted on the jaws of the machine, one end movable and the other stationary, the machine was operated at a constant rate of extension. Throughout the procedure, the force (F) applied and the elongation (ΔL) of the test piece are measured.

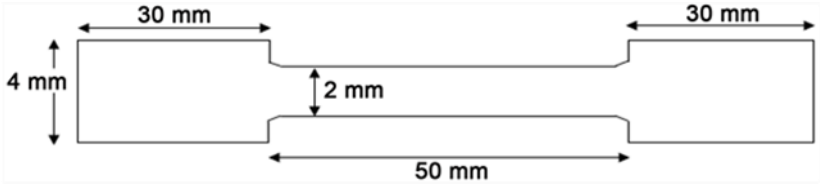

Figure 3: Dimension of tensile test sample

2.6 Microstructural Analysis

This was done via optical and scanning electron microscopy. Specifications outlined in ASTM E407 and ASTM A247 standard for nodularity and nodule count analysis was adopted for optical microscopy. Sectioning of the samples was done, after which they were mounted for grinding and polishing. Etching was done using 2% nital solutions in order to reveal the phases present under the microscope. The estimation of the nodularity rating was done in according to Equation 1.
For the SEM analysis, the samples were placed in the viewing chamber of the SEM machine located at National Geosciences Research Laboratories, Nigerian Geological Survey Agency, Kaduna State.

$$Nodularity\ \% = \frac{Area\ (number) of\ acceptable\ particles}{Area\ (number) of\ all\ particles}\ \text{x}\ 100 \quad (1)$$

2.7 X-Ray Diffraction (XRD) analysis

The phases present in all the samples were determined via XRD. X-rays were directed onto the samples, and the resulting diffraction patterns were recorded for Braggs angle between 10° and 70°. The diffraction patterns were then analysed in order to determine the crystal structure and phase composition of the material.

3. RESULTS AND DISCUSSIONS

3.1 Elemental Composition of As Cast TWDI Samples

The elemental composition of the as cast TWDI sample is shown in Table 1, the results shows that cast TWDI sample has the correct compositions. The TWDI samples designation with respect to the soaking time intervals is recorded in Table 2.

Table 1: Elemental composition of As-cast TWDI

Elements	Fe	C	Si	Mn	P	S	Mg
Wt. % Conc.	94.01	3.28	2.12	0.43	0.03	0.05	0.038

Table 2: Sample designations with their soaking time

TWDI Samples	Sample 1 (As cast)	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7
Soaking time	0mins	5mins	10mins	15mins	20mins	25mins	30mins

3.2 Microstructural Characterization

3.2.1 Optical and scanning electron micrographs (OM and SEM)

The micrographs of sample 1 (as cast) for unetched and etched are shown in Figures 4a and 4b, whereas Figures 5 -10 shows the OM micrographs of samples 1 to 7 in their etched condition. The SEM micrographs for samples 1, 3, 5 and 7 are shown in Figures 11-14 respectively.

The nodularity and nodule count of the as cast sample was estimated according to the specifications outlined in ASTM E407 and A247, the result is shown in Table 3. These values remained the same as the heat treatment progressed. The microstructure of the as cast sample composed of large volumes of pearlite phase with some proportion of the ferrite phase in the matrix. Large proportions of eutectic carbide precipitation were also observed; this resulted from fast cooling evident in TWDIs. These carbide precipitates were needle/ platelike in structure indicating the shape of the primary carbides of M_3C type. These carbides are also present in large volumes as seen in Figure 11 from the SEM micrographs.

At the soaking time of 5 minutes (sample 2) as seen in Figure 4, a slight reduction in the carbide precipitates was observed, though this soaking time was not sufficient to bring about significant dissolution of the carbide phase. As the soaking time progressed from 10, 15, 20 and 25 minutes, it was observed that reduction of the carbide phase was proportional to the soaking times as seen in Figures 6, 7, 8 and 9 respectively. The large proportion of pearlite phase still remained the dominant matrix phase. This is also evident in Figures 12 and 13. During the annealing treatment process, the graphite nodules acted as carbon sinks for the dissolved carbide phase, the small distance between the graphite nodules favoured this dissolution. This observation is in consonance with that observed by the researchers in [14].

The microstructure of the sample with 30 minutes soaking time (sample 7) showed little or no carbide precipitates as shown in Figures 10 and 14 for OM and SEM micrographs respectively. Matrix type was also predominantly the pearlite phase, also ferrite phase larger than that observed in the as cast phase. It was observed that as soaking time increased, the ferrite phase increased slightly, graphite nodule became more rounded and carbide phase dissolve at a faster rate, this observation was in consonance with the trend observed in [15]. The adopted heat treatment process has successfully reduced the carbide precipitates that form during solidification of the TWDI sample.

Table 3: Nodule count and nodularity of sample

S/N	**Specimen**	**Nodularity (%)**	**Nodule Count (nodules/mm^2)**
1	Sample 1	88	546

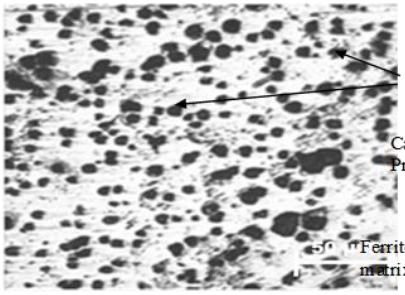

Figure 4a: Sample 1 (As cast Unetched)

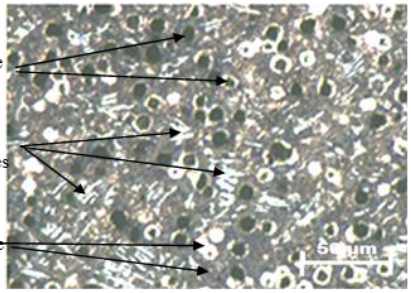

Figure 4b: Sample 1 (As cast Etched)

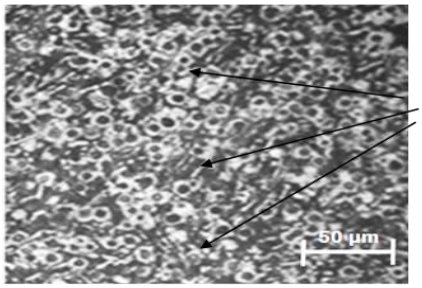

Figure 5: Sample 2 etched

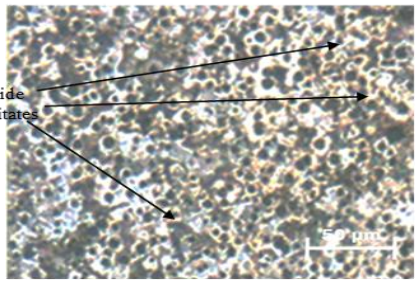

Figure 6: Sample 3 etched

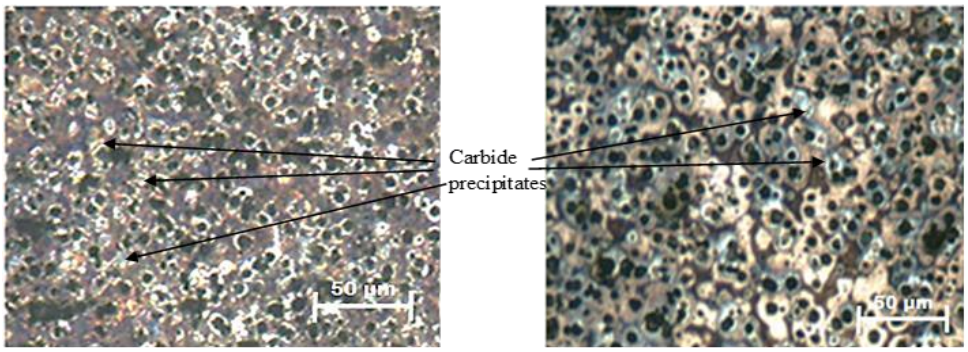

Figure 7: Sample 4 etched

Figure 8: Sample 5 etched

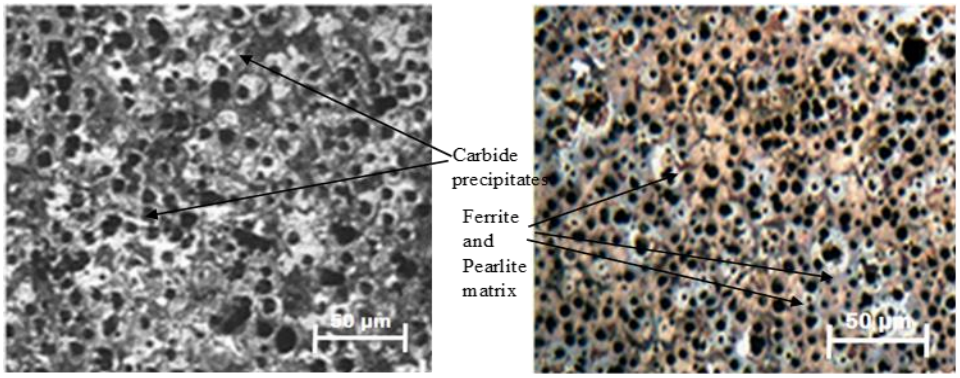

Figure 9: Sample 6 etched

Figure 10: Sample 7 etched

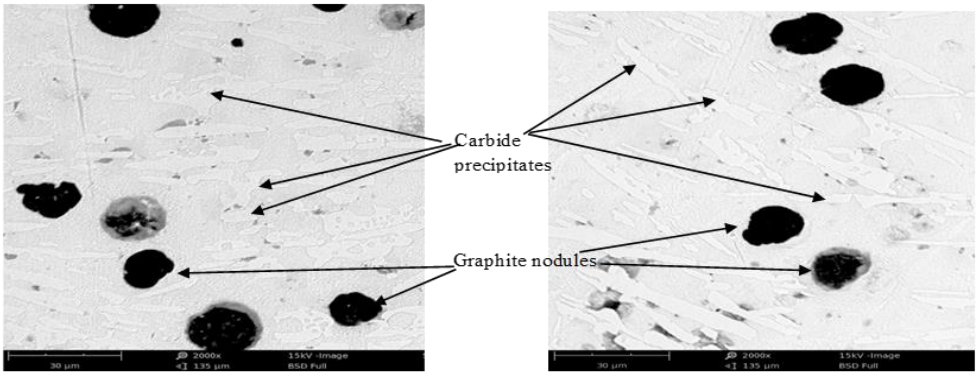

Figure 11: Sample 1 (As cast)

Figure 12: Sample 3

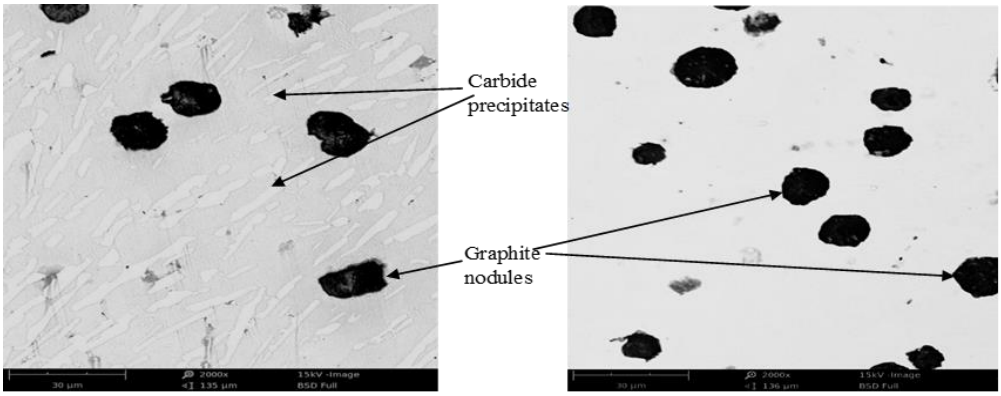

Figure 13: Sample 5

Figure 14: Sample 7

3.3 Mechanical Tests

3.3.1 Ultimate tensile strength (UTS)

The ultimate tensile strength (UTS) plot of all the samples is shown in Figure 15. The as cast sample gave the highest value of 596 MPa, then there was a progressive reduction in heat-treated samples tensile strength values as soaking times were increased. This is attributed to the reduction in the carbide precipitation as the increased times led to dissolution of the carbide precipitates. Also, the reduction in volume of the pearlite phase and the slight increase in the volume of the ferrite phase can be attributed to the reduction in the UTS values. The sample soaked for thirty (30) minutes gave the lowest UTS value.

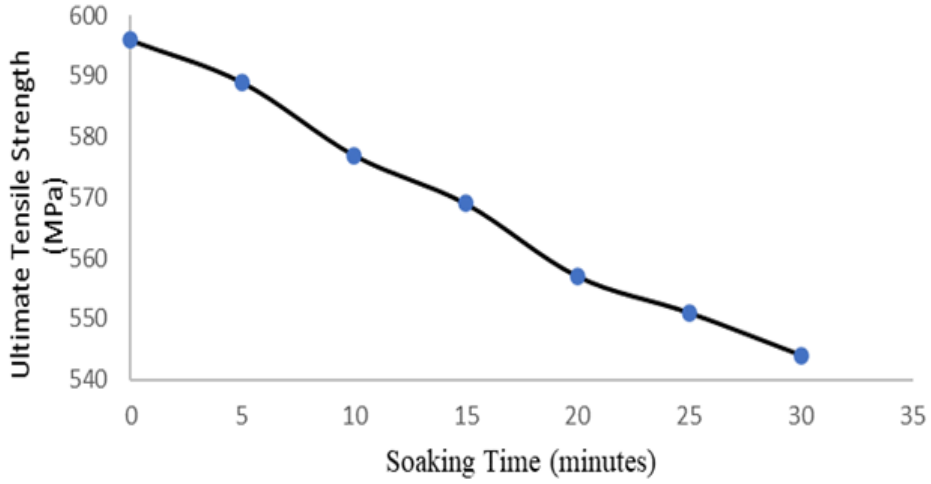

Figure 15: Plot of ultimate tensile strength of samples versus soaking times

3.3.2 Percent elongation

The plot of percent elongation with the various soaking times is shown in Figure 16. This value increased with increase in soaking times. This is attributable to the progressive reduction in carbide precipitates and also the reduction of the large volumes of pearlite phase and the slight increase in the proportion of ferrite phase. The as cast phase gave the value of 4.3%, whereas soaking times of 5, 10, 15 20, 25 and 30 minutes gave 4.7%, 5.8%, 6.5%, 7.2%, 8.1% and 8.7% respectively. This trend showed that the annealing treatment adopted reduced the carbide phase significantly to positively impacted on the percentage elongation values.

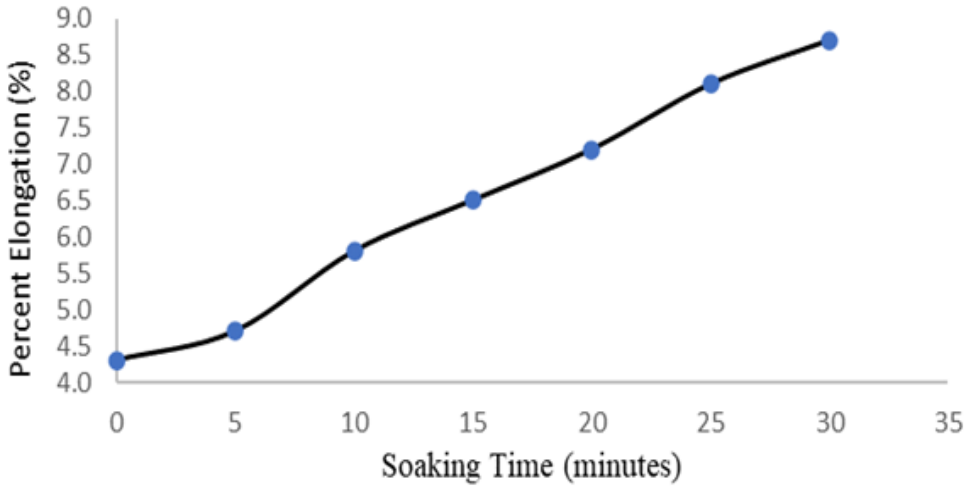

Figure 16: Plot of percent elongation of samples versus soaking times

3.3.3 Vickers hardness

The plot of Vickers hardness values for the samples with the various soaking times is shown in Figure 17. This value decreased with increase in soaking times. This is attributable to the progressive reduction in carbide precipitates and also the reduction of the large volumes of pearlite phase and the slight increase in the volume of ferrite phase. The large volume of the carbide phase and pearlite phase gave the as cast phase its high hardness value of 291.6 Hv, which was the highest

hardness value recorded for all the samples, whereas the sample soaked for 30 minutes gave 243.5 Hv, which was the lowest hardness value.

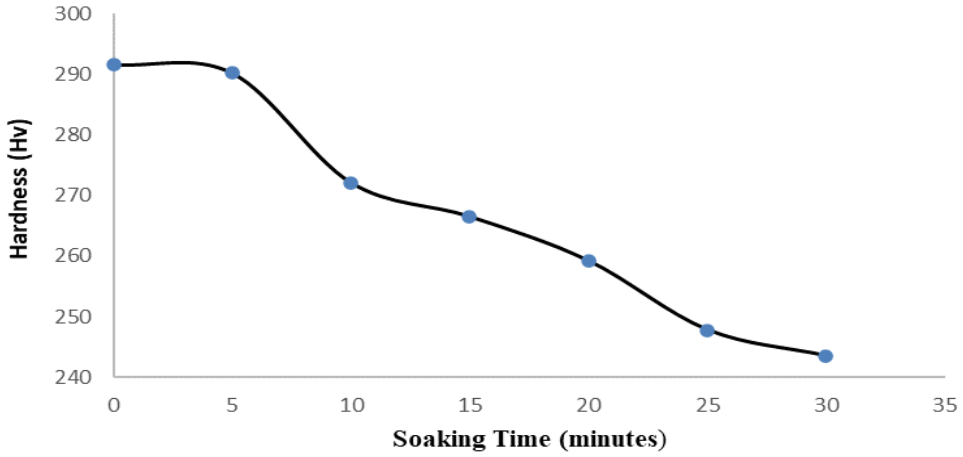

Figure 17: Plot of Vickers hardness of samples versus soaking times

3.4 X ray Diffraction Analysis (XRD)

The X-ray diffraction patterns were done for some selected samples namely the as-cast, 10-, 20- and 30-minutes-soaked samples i.e., samples 3, 5 and 7 in order to study the phases present in the microstructure as the heat treatment progressed, these patterns are shown in Figure18. The predominant phase of all the samples consisted of ferrite (α-Fe), graphite (C) and cementite (Fe_3C), which is in consonance with phases observed in the study of [16]. Cementite with peak at 2θ angle of 44° (220) is the main phase observed for as cast and 5 minutes-soaked sample (Samples 1 and 2). The peak observed in plane (100) at Bragg angle of 42° for samples 1 and 2 show the presence of graphite, showing that carbon is one of the main elements in TWDIs. BCC ferrite (α-Fe) peak of low-intensity in (200) plane at 66° for 2θ was observed in the pattern of all samples. The presence of ferrite and cementite peaks shows that pearlite phase is present in the matrix as pearlite comprises of alpha-ferrite (α-Fe) and cementite (Fe_3C).

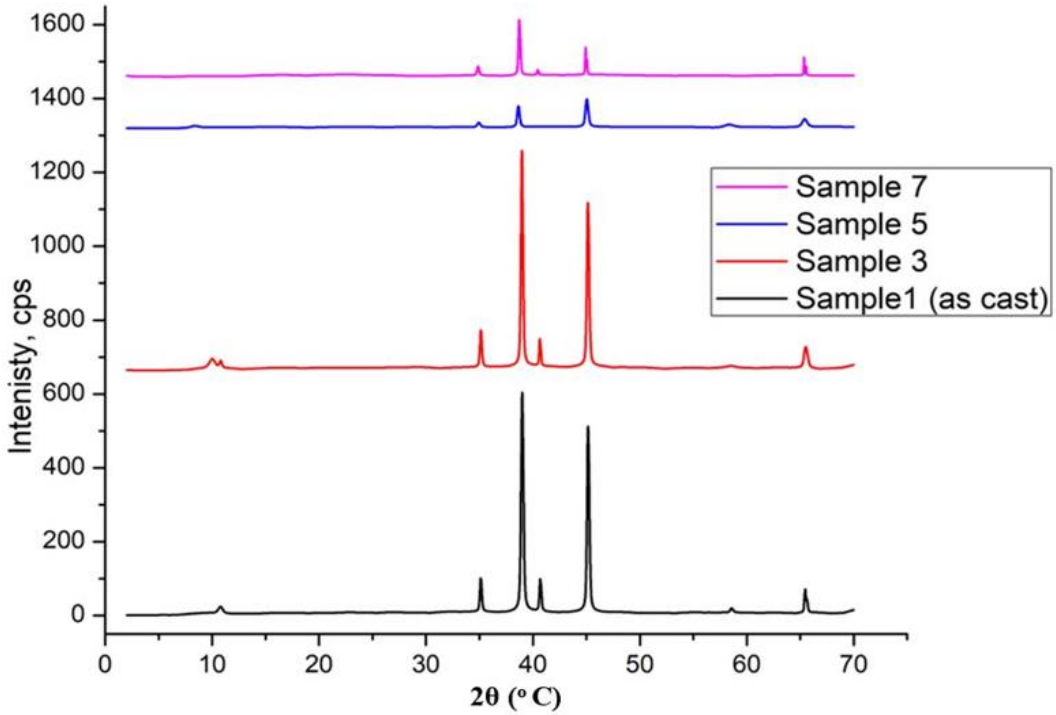

Figure 18: X-Ray diffraction patterns for samples 1, 3, 5 and 7

Table 4: Table of results (summary)

TWDI Sample	Soaking time (mins)	UTS (MPa)	Percent elongation	Vickers hardness (Hv)
Sample 1 (As cast)	0	596	4.3	291.6
Sample 2	5	589	4.7	290.2
Sample 3	10	577	5.8	272.1
Sample 4	15	569	6.5	266.5
Sample 5	20	557	7.2	259.2
Sample 6	25	551	8.1	247.8
Sample 7	30	544	8.7	243.5

4. CONCLUSION

Heat treatment of As-cast TWDI samples containing large volume of carbide precipitation within its microstructure was examined in this study. Sample 1 (as-cast) exhibited a predominantly pearlite phase with reduced ductility and a moderate nodularity and nodule count ratings. Dissolution of carbide precipitates was achieved by employing 10, 15-, 20-, 25- and 30-minutes soaking times to the cast TWDIs. Microstructure and mechanical property improvements were observed as soaking times were increased from 10-30 minutes. The annealing heat treatment process conducted has shown to be effective in reducing or eliminating carbide phases.

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