FRACTURE TOUGHNESS AND MICROSTRUCTURE OF HEAT-TREATED 0.22% CARBON STEEL

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ABSTRACT
Effect of heat treatment on fracture toughness and microstructure of 0.22% carbon steel has been investigated. Samples of 0.22% C steel were subjected to annealing and normalising heat treatments at a temperature of 900 °C. Tensile tests were then conducted on standard tensile and circumferential notched tensile specimens of both as-received and heat treated steels. Microstructural investigations were also performed on the specimens. Heat treatment resulted in changes in the microstructure and fracture toughness. It was observed that the as-received sample had a higher value of fracture toughness than the heat-treated samples due to finer morphology of ferrite and inter-lamellar spacing of pearlite than the annealed and normalized samples. The fracture toughness results obtained using the CNT test samples were found to be valid (in plain strain condition) and in close agreement with data available in literature.

Keywords: CNT, fracture toughness, carbon steels, heat treatment.

INTRODUCTION
Despite the introduction of composites in recent years, metals and alloys still remain important in structures because of their strength, stiffness, toughness and tolerance of high temperatures. They are however susceptible to failure and chemical attack if not given required treatment. Hence, heat treatment of metals is done to modify their microstructure and as a result, yield property improvement (Agboola et al., 2013).
The mechanical properties of any structural metallic material, with certain chemical composition, are a function of its microstructure. Heat treatment of steels is a common practice for manipulating microstructure of steels. Low carbon steels are usually heat-treated to modify their grain size which, according
to Hall-Petch equation, controls the strength of metallic materials (Jahjah & Dobranszky, 1991). Several mechanical tests are available to assess the mechanical properties of heat-treated steels to ascertain the extent to which they have been affected. A major test procedure is the tensile test- from which important properties of material such as tensile strength and ductility can be estimated. Another material property of great importance that could be estimated from tensile test is fracture toughness. The property is used in an analogous manner as the yield strength in the strength of materials approach. Fracture toughness is a controlling parameter used in machine design to avoid catastrophic brittle failure (ASTM, 2004). It measures the ability of a material containing a crack to resist fracture (Dieter, 1988) and is one of the most important properties of any material for virtually all design application. Fracture occurs when the stress intensity factor K reaches a critical value $K_{IC}$ as the crack grows. The mode-1 stress intensity factor at the onset of rapid crack propagation under plain-strain condition is known as plain strain fracture toughness ($K_{IC}$). It is a material property (Bayram et al., 2002).

A number of researches have confirmed fracture toughness could be estimated from the tensile test procedure (although with a sample known as the Circumferential Notch Tensile specimen, CNT) (Nath and Das, 2006; Alaneme, 2011). This test procedure for measuring fracture toughness is rapid, cost effective and reliable (Alaneme, 2011).

In the present work, effect of heat treatment on fracture toughness and microstructure of low carbon steel has been investigated.

**EXPERIMENTAL PROCEDURE**

**Materials**

The material used for this study is low carbon steel sourced from Yankarfe, Zaria, Nigeria. Chemical analysis of the steel was carried out at National Metallurgical Development Centre (NMDC) Jos and is presented in Table 2.1.

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt. (%)</td>
<td>0.22</td>
<td>0.24</td>
<td>0.73</td>
<td>0.09</td>
<td>0.04</td>
<td>0.03</td>
<td>0.08</td>
<td>0.02</td>
<td>98</td>
</tr>
</tbody>
</table>

**2.2 Methods**

The experimental steps are:

(i) Sample preparation
(ii) Heat treatment
(iii) Microstructural analysis
Sample Preparation

12mm low carbon steel samples was machined to standard dimensions for tensile test. Two sets of tensile test specimens were machined; one, v-notched centrally (as shown in Figure 2.1) and the other, un-notched. The dimensions of the two geometries are as follows:

Specimen with notch (CNT)
- Diameter of the grip length (D): 7mm
- Diameter of the gauge length (d): 5mm
- Gauge length (l₀): 30mm
- Grip length (l): 10mm
- Notch diameter (d₀): 4mm
- Notch angle (α): 60°

Specimen without notch
- Diameter of the grip length (D): 7mm
- Diameter of the gauge length (d): 4mm
- Gauge length (l₀): 30mm
- Grip length (l): 10mm

Figure 2.1: Schematic of circumferentially notched round bar.

Table 2.2: Number of experimental samples

<table>
<thead>
<tr>
<th>Test</th>
<th>Control</th>
<th>Annealed</th>
<th>Normalized</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metallography</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>Tensile (notched)</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>6</td>
</tr>
<tr>
<td>Tensile (unnotched)</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>6</td>
</tr>
</tbody>
</table>

Heat treatment

The total number of samples machined for annealing and normalizing heat treatments are shown in Table 2.2. The machined test samples were placed in the furnace for heat treatment operation. The samples were heated to austenitizing temperature of 900°C and allowed to soak for 25 minutes; after which the furnace was switched off and a set of samples taken out to cool in air (i.e. normalizing),
while the other was left to cool in the furnace (i.e. annealing) to room temperature.

**Metallography**

Samples for metallography (control, annealed and normalised) were prepared by successive steps of grinding, polishing and etching. The samples were ground on a water lubricated silicon carbide abrasive paper of 180, 240, 320, 400, 600 and 800 grit sizes in succession. Polishing was carried out on 15cm rotating discs universal polishing machine with a synthetic velvet polishing clothes impregnated with 1um alumina paste. The specimens were then etched with 2% nital solution by swabbing with a cotton soaked in the etchant. Finally, the microstructures of the steel samples were visualized under a metallurgical microscope.

**Mechanical testing**

The mechanical tests that were carried out in this work to determine the mechanical properties of the untreated and heat treated steel samples and the procedures are as follow:

**Tensile test**

Room temperature uniaxial tension tests were performed on CNT and tensile samples using a 50kN Instron universal testing machine at room temperature. The specimen was loaded monotonically in tension with a crosshead displacement rate of 5mm/min until failure. The procedure adopted was in conformity with ASTM E8M (Philadephia, ASTM 1991). Two repeat tests were performed for each test condition to guarantee reliability of the data generated. The maximum tensile load at fracture was recorded for each test specimen and the fracture toughness calculated by substituting fracture load, D and d in Equation 2.1. The tensile properties evaluated from the stress-strain curves developed from the tension test are: ultimate tensile strength ($\sigma_u$), yield strength ($\sigma_y$), strain to fracture ($\epsilon_f$) and elastic modulus (E). Data from test of Circumferential Notch Tensile, CNT Specimen and tensile specimen were used to evaluate fracture toughness of the sample.

**Fracture toughness, $K_{IC}$**

Fracture toughness is determined by using standard specimen geometries such as compact- tension (CT) specimen or single edge notch bend (SENB) or three point loaded bend specimen which are standardized by ASTM (Dieter,1988). In
these techniques, specimen preparations and test are quite tedious and time consuming. This work explains a simple test methodology based on fracture mechanics approach using the Circumferential Notched Tensile (CNT) specimen (Figure 2.1). Specimen preparation and fatigue pre cracking which is quite simple, consuming less material and machining time. This CNT specimen, being small scale specimen is widely useful in situations where large volume of specimen material is not easily available. It was also observed that the circumferential crack propagates radially inwards in tensile loading which make them suitable for studying the impact of microstructure on fracture toughness of metal unlike unique crack propagation towards one direction in case of CT or SENB specimens (Alaneme, 2011). This test methodology is simple, reliable, rapid and cost effective results in a valid fracture toughness value quite close to the true fracture toughness. The advantage of the CNT specimens for fracture toughness evaluation include: 1) attainment of plain strain crack loading conditions with smaller specimen dimension in comparison with CT specimen (Alaneme, 2011); 2) radial symmetry which make them suitable for studying the impact of microstructure on the fracture toughness (Nath and Das, 2006); 3) ease of machining to desired test configuration (Alaneme, 2011); 4) ease of testing using simple testing facilities like the tensometer (Nath and Das, 2006).

Circumferential notch tensile, CNT tensile specimen geometry can be considered as a standard test specimen in fracture toughness test.

The expression used for determining fracture toughness (\(K_{IC}\)) from round notched tensile specimen (Dieter, 1988) is given below:

\[
K_{IC} = \left(\frac{P_f}{D^{3/2}}\right) \left[1.72 \left(\frac{D}{d}\right)^{0.5} - 1.27\right]^{1/2}
\]

Where \(P_f\) is the fracture load, \(D\) is the diameter of the specimen, and \(d\) is the diameter of the notched section (Figure 2.1). The assumption taken while formulating above expression is that the specimen retains its elastic behaviour until fracture occurs. This relation is valid for the \(D/d\) ratio between 1.00 and 1.25 (Nath and Das, 2006). The required plain strain state is, even in the case of a round-notched tensile specimen, created only if the diameter of such specimen is above a certain minimum. For round notched and pre-cracked tensile specimens, the following condition is to be fulfilled:

\[
D \geq \left(\frac{K_{IC}}{\sigma_{YS}}\right)^2
\]

Where \(\sigma_{YS}\) is the yield stress. An additional requirement is that the length of specimen \(L\) be at least \(4D\) (Alaneme, 2011).

The notched tensile strength (\(\sigma_{NTS}\)) were also evaluated following standard procedures in accordance with (Bayram et al, 2002). The notched tensile
strength value ($\sigma_{NTS}$) obtained for each treatment condition was utilized to validate the reliability of the fracture toughness results obtained from Equation 2.1, using the relation (Nath and Das, 2006):

$$K_C = 0.454\sigma_{NTS}(D)^{\frac{3}{2}}$$  

2.3

RESULTS AND DISCUSSION

Effect of heat treatment on microstructure
Plate 1: Microstructures of (a) as-received (b) annealed and (c) normalized samples at 100X

The micrographs in plate 1(a), (b) and (c) at magnification 100X reveal the microstructures of the as-received, annealed and normalised specimens respectively. The microstructure of the as-received specimen consists mostly of ferrite and increasing amount of cementite (iron carbide) in a laminar structure called pearlite. Black regions depict pearlite and white regions depict ferrite. Ferrite is clearly observed as matrix and pearlite is the dispersed phase (Callister, 2007).

The microstructure of annealed specimen, as can be seen in plate 1(b), consists of proeutectoid pearlite mixed with ferrite and cementite. The main difference between the as-received and annealed specimen is, the pearlite colonies and the ferrite are coarser in the annealed specimen as compared to as-received specimen. This is because as slow cooling is done in annealing, the steel of 0.22% C is transformed from a single phase of austenite to pearlite structure, a lamellar or layered structure of two phases: ferrite and cementite (Dieter, 1988).

As in the case of annealing, the microstructure of the normalized specimen as shown in plate 1(c) also consists of proeutectoid pearlite also mixed with ferrite and cementite, but in normalizing, since cooling rate is higher, transformation of austenite takes place at much lower temperature when compared with
annealing. As a result, the transformation product, pearlite, is finer with lower inter-lamellar distance between the two neighbouring cementite plates. This is in agreement with the findings of Mahbour et al. (2016).

**Effect of heat treatment on tensile strength of 0.22% C steel**

![Graph showing tensile strength of as-received, annealed, and normalized samples.](image)

**Figure 3.2: Tensile strength of as-received and the heat treated samples**

Figure 3.2 shows the effect of heat treatment on tensile strength of 0.22% C steel. Tensile strength (σ_u) values for as-received, annealed and normalized specimens were observed to be 664.21MPa, 450.08MPa and 501.35MPa respectively. The lowest tensile strength for the annealed specimen can be associated with the formation of coarser pearlite colonies in the ferrite matrix which allows free movement of dislocation, in contrast to the normalised and as-received where dislocation motion is constrained by the fine microstructure which results in stronger material (Callister, 2007). The increase in tensile strength of the normalized sample as compared to the annealed was due to faster cooling rate and due to it, the transformation product, pearlite is finer with lower interlamellar distance between the two neighbouring cementite phases and according to Hall-Petch equation, a fine-grained material (one that has small grains) is harder and stronger than one that is coarse grained. This agrees with the findings of Fadare et al. (2011).
3.3 Effect of heat treatment on the fracture toughness of 0.22%C steel

Figure 3.3: Fracture toughness of as-received and treated CNT samples

Figure 3.3 shows the effect of heat treatment on fracture toughness ($K_{IC}$) of 0.22%C steel. Fracture toughness ($K_{IC}$) values for as-received, annealed and normalized specimens were found to be 24.38MPa($m^{1/2}$), 17.04MPa($m^{1/2}$) and 19.55MPa($m^{1/2}$) respectively. Fracture toughness ($K_{IC}$) value, calculated using CNT specimens, is highest for as-received steel as compared to annealed and normalised steel due to finer morphology of the as-received. The reliability of the results was assessed by determining if nominal plain strain condition was achieved for the CNT specimen dimension (D) of 5mm utilized for the test. It was observed that the minimum specimen thickness required to attain plain strain conditions when the relation $D \geq \left(\frac{K_{IC}}{\sigma_{YS}}\right)^2$ (Equation 2.2) was utilized is less than 5 mm for all treatment conditions. Thus the results are reported as plain strain fracture toughness under tensile mode ($K_{IC}$). The notch tensile strength $\sigma_{NTS}$ has been reported to give a good measure of fracture toughness by the relation $K_C = 0.454 \sigma_{NTS}(D)^{1/2}$ (Equation 2.3). The fracture toughness results from the CNT test were valid (in plain strain condition). The value of 24.38MPa($m^{1/2}$) obtained for the as received 0.22%C steel corresponds with
CONCLUSION
From the results obtained in this study, it can be concluded that:

1. Heat treatment resulted in modified microstructures, with the annealed specimen having larger grains; although the treatment did not improve the mechanical properties of the carbon steels.

2. The reliability of the circumferential notch tensile test was established by checking the values obtained with some empirical relations. And 24.38 MPa \( m^{1/2} \) obtained for the as-received 0.22% C steel is close to 25 MPa \( m^{1/2} \) reported for plain strain fracture toughness \( (K_{IC}) \) of low carbon steel in literature.

3. Summarily, the results indicate that the best combination of tensile strength and fracture toughness is achieved for as received sample due to finer morphology of ferrite and finer interlamellar spacing of pearlite in the as-received condition which ensured good mechanical properties.

REFERENCES
