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THERMOCHEMICAL HEAT TREATMENT OF MEDIUM CARBON STEEL (Nst 60-2) IN THE ATMOSPHERE OF NATURAL NITROGENOUS MATERIALS

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ABSTRACT

Thermo-chemical heat treatment behaviour of medium carbon steel grade (Nst 60-2) is investigated using nitrogenous materials. In this study the effect of carbonitriding and nitriding on the formation and decomposition of martensite and other phases of steel are investigated. Tensile specimen with and without notch were produced and subjected to Nitriding with Bean Leaves (NIBL), Nitriding with Cow Dung (NIBL), Carbonitriding with Charcoal and Bean Leaves (CNCBL) and Carbonitriding with Charcoal and Cow Dung (CNCCD). Microstructural examinations and chemical analyses of the samples were conducted. The ductility, ultimate tensile strength and yield strength of the heat treated samples were determined from their stress-strain curves. The Ultimate Tensile Strength (UTS) was observed to be 42% higher for NICD specimen as a result of the grain re-arrangement, followed by that of CNCCD specimen with 36% then CNCBL with 27% and NIBL with 24% increase compared with the control specimen which had UTS of 1248.56 N/mm^2. Experimental results were analyzed graphically to reveal the variations that complemented the microstructures. The case of the steel became hardened with soft core, thereby making the steel component to be tough and wear resistant. The average UTS for thermo-chemically heat treated specimens are the same with or without a notch, but the average Yield Stresses (YS) are 2.3-4.2% lower for specimens without notch.

KEYWORDS: Pearlite; Nitrogenous materials; Ultimate Tensile Strength; Carbonitriding; Nitriding.

1. INTRODUCTION

Thermo-chemical heat treatment involves the diffusion of carbon, nitrogen and, less commonly, boron to pre-determined depths into the steel surface. These elements may be added individually or in combination, resulting in a surface with desirable properties and of different composition to the bulk. Thermo-chemical heat treatment processes produce a hard, wear-resistant surface or case over a strong, tough core. The principal forms of thermo-chemical heat treatment processes are carburizing, nitro-carburizing, cyaniding, nitriding, carbonitriding and boronising. Only ferrous metals are suitable for thermo-chemical heat treatment. Thermo-chemical heat treatment is ideal for parts that require a wear-resistant surface and must be tough enough internally to withstand heavy loading. In thermo-chemical heat treatment, the surface of the steel is chemically changed by adding high carbide or nitride content but the core remains chemically unaffected. When heat- treated, the high-carbon surface responds to hardening and the core toughens.

Changes such as improved wear properties, corrosion resistance and improved fatigue strength are also obtained after thermo-chemical heat treatment. In this study, the medium carbon steel samples were collected from Dana Steels Rolling Mill limited, Katsina, Nigeria. During carbonitriding and nitriding in salt bath and pack carburizing, the surface of medium carbon steel is enriched with carbon and nitrogen, usually at temperatures between 850°C and 950°C. Heating molten cyanide salts to the reaction temperature releases carbon by decomposing the cyanide.

The nitriding and carbonitriding processes used in this investigation involved addition of carbon and nitrogen to the surface of the medium carbon steel by exposing it to carbon and nitrogen rich atmosphere made up of charcoal, bean leaves and cow dung.

2 LITERATURE REVIEW

According to Woehrle *et al.* (2012), nitriding changes, primarily, the surface related properties of a material. However, the presence of nitrided case also affects properties of the material beneath the nitrided case and the entire component. A comparative study by Campagna *et al.* (2011) confirmed that both carbonitriding and nitrocarburizing processes develop compressive stress and are associated with the size and shape distortion. However, nitrocarburizing causes lower compressive stress and size/shape distortion, as is the case for SAE 1010 steel.

Carlos (2011) found that nitriding is a thermo-chemical process that gives greater hardness to the surface of parts, providing greater resistance to external fatigue and friction. It was reported that nitriding is often carried out at or below the tempering temperature of steels and they can be hardened prior to nitriding and the nitriding can also be used as a temper. The report also found that nitriding is intended to obtain parts with higher surface hardness, to increase the resistance against wear, fatigue, corrosion, and heat.

Carlos (2011) reported that nitriding process can be carried out in furnaces or salt baths controlled atmosphere (gas) after which the surface of the carbonitrided layer acquires hardness and wear resistance. It was suggested that the process temperature ranges from 705ºC to 900ºC for an exposure period of two hours, after which, the pieces are cooled in water or oil. Using this method it is possible to get a layer with thickness from 0.07 to 0.7 mm.

Ndliman (2006) investigated the mechanical properties of a medium carbon steel heat treated at 850˚C and quenched in both water and oil. It was found that the tensile strengths of the heat treated samples were higher than that of the standard AISI C1035 steel sample. The sample quenched in water showed a higher strength than the one quenched in oil. Martensitic structure, which has a detrimental effect on toughness, is also produced during continuous water quenching (Madariage, 1999).

2. MATERIALS AND METHODS

3.1 Materials

A sample of medium carbon steel (Nst 60-2) (See Appendix for composition) was obtained from Dana Steels Rolling Mill Ltd, Katsina, Nigeria. Tensile specimens with and without notch were produced from the steel and were subjected to various forms of thermo-chemical heat treatment processes (carbonitriding and nitriding) using natural nitrogenous materials. The specimens were subjected to controlled heating and cooling to effect changes in metallurgical and mechanical properties. The ductility, ultimate tensile strength and yield strength of the heat treated samples were obtained from their stress-strain curves.

3.2 Methods of analyses

To evaluate the effect of thermo-chemical heat treatment processes on the medium carbon steel, the investigation was carried out thus;

(i) Preparation of the tensile specimens with and without notch from Nst 60-2 medium carbon steel (0.35 – 0.42% carbon content).

(ii) Thermo-chemical heat treating of the specimen.

(iii) Metallographic examination of the specimens.

(iv) Tensile test of the medium carbon steel specimens.

2.3 Thermo-chemical heat treatment process

Standard heat treatment procedures were adopted (Totten, 2007) to heat treat the medium carbon steel which will be explained under carbonitriding and nitriding processes. Different samples were prepared from the medium carbon steel for each of the operations (carbonitriding and nitriding) as outlined below. A total number of twenty four (24) specimens were heat treated for the study.

3.3.1 Carbonitriding: Carbonitriding was carried out on twelve (12) specimens by heating them slowly at 850°C in an electric furnace. The specimens were pickled and cleaned before burying them in carbon and nitrogen-rich mixture. These natural sources of carbon and nitrogen were packed with molasses which act as a binder that enabled the specimen to be thoroughly heat treated without any loss of carbon and nitrogen at austenizing temperature and carburizing time. The bottom of the carburizing container was covered with charcoal and beans leaves and charcoal and cow dung to about 20-30mm deep before burying the specimens for CNCBL and CNCCD heat treatments respectively. The carburized containers were placed in an electric furnace and heated to a temperature of 850°C and this temperature was maintained for duration of 3hours for all the material to transform into austenite. The specimens were then allowed to cool in still air.

Meanwhile another set of the specimens which were not heat treated were taken directly for the tensile tests to serve as control samples.

2.3.2 Nitriding Process: A nitriding process was carried out on twelve (12) specimens by heating them slowly at 750°C in an electric furnace for NIBL and NICD heat treatments. The specimens were pickled and cleaned before burying them, separately, in bean leaves and cow dung nitrogen – rich mixture. These natural sources of nitrogen were packed with molasses which act as a binder that enabled the specimen to be thoroughly heat treated at austenizing temperature and carburizing time without any loss of nitrogen.

The bottom of the carburizing container was covered with beans leaves and cow dung respectively to about 20-30mm deep before burying the specimens. The carburized containers were placed in an electric furnace and heated to temperature of

750°C and this temperature was maintained for duration of 3hours for all the material to transform into austenite. The specimens were then allowed to cool in still air. Some of the specimens were tempered by reheating to 200^0C for 3 hrs. The carburized component was later prepared for metallographic examination.

2.4 Tensile test of steel specimens

Tensile test was conducted using a standard test specimen, in accordance with ASTM E8 standard for tensile test. The specimen was mounted on TQ SM 100 Standard Universal Material Testing Machine as shown in Plate1 below. The load was increased at a constant rate by a hydraulic action causing elongation, reduction of cross sectional area and eventual fracture of the specimens. The tensile tests were conducted with heat treated specimens and the results were compared with those of non-heat treated specimens.

The initial cross section area, final cross section area, original diameter, fracture load, Yield Strength and Ultimate Tensile Strength for all the specimens were recorded and also used for further analysis. Similar test procedure was also adopted for specimens' carbonitrided with charcoal and cow dung, nitriding with bean leaves and cow dung and control samples respectively.

3.5 Metallographic examination of steel samples

A sample from each group was prepared for micro-structural metallographic examination. The specimens were first ground using a grinding machine and then manually polished to mirror finishing using 240 and 320 grades of emery paper. After polishing the surface was etched using 5% Nital. Micrographic examination of the prepared samples was conducted using Scanning Electron Microscopic equipment (SEM) Model No.JEDL-120AUS (100 magnification) in accordance with ASTM E107 standard.

3. RESULTS AND DISCUSSION

4.1 Tensile Strength Test Results

Tensile tests were carried out on the steel specimen with and without notch as shown in Tables 1 and 2.

Ductility: The ductility of metals is usually considered to be characterized by the elongation of the gauge length of the specimen during a tensile test and by the reduction in area of the cross section at the fracture (Timoshenko, 1988):

$$Ductility = \frac{change\ in\ length}{original\ length} \ldots \qquad (1)$$

For Carbonitrided with charcoal and bean leaves combined (Carbonitrided A), the ductility was found to be 16%.

Table 1: Tensile Test Results of Thermo-Chemically Heat Treated Specimens With Notch (For Bridgman correction factor (B) = 1.00)

Thermo-chemical heat Treatment Processes	Ductility (%)	Average Ultimate Tensile Strength (N/mm^2)	Average Yield strength (N/mm^2)	True UTS (N/mm^2)	True Yield Strength (N/mm^2)
CNCBL	16	1587.76	1083.48	1305.13	890.62
CNCCD	13	1696.02	1329.01	1394.18	1092.45
NIBL	15	1551.67	1160.05	1275.47	953.56
NICD	10	1768.19	1240.26	1453.45	1019.49
Control (as received)	19	1248.56	1055.05	1026.32	867.25

Table 2: Tensile Test Results Of Nst 60-2 Of Thermo-Chemically Heat Treated Specimens With Notch (For B=0.876)

Thermo-chemical heat treatment processes	Ductility (%)	Average UTS (N/mm^2)	% increase in UTS	Average YS (N/mm^2)	% increase in YS
CNCBL	16	1587.76	27.2	1046.48	4.6
CNCCD	13	1696.02	35.8	1299.08	28.6
NIBL	15	1551.67	24.3	1118.65	10.7
NICD	10	1768.19	41.6	1190.82	17.9
Control (as received)	19	1248.56	-	1010.39	-

4.1 Micrographic examination:

The observations made under the Scanning Electron Microscope (SEM) show the microstructure of the samples. In Plate 2, martensite partly transformed into ferrite (white) and cementite. In Plate 3, Carbon precipitated with martensite (black) and bainite (dark gray) region forming needle like structure with some ferrite and cementite. In Plate 4, fine-grained martensite (black), ferrite-pearlitic structure and some untransformed austenite (gray white). In Plate 5, the steel is made up of ferrite (white) + pearlite (black)

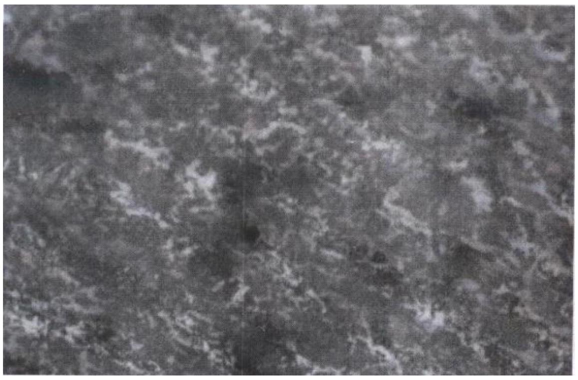

Plate 2: Microstructure of Nst 60-2 CNCBL Specimen *x 100mag* from transverse orientation

Plate 3: Microstructure of Nst 60-2 CNCCD Specimen *x 100mag* from transverse orientation

Plate 4: Microstructure of Nst 60-2 NIBL Specimen *x 100 mag* from transverse orientation.

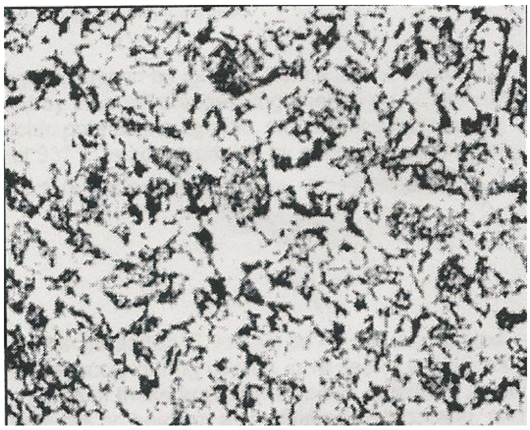

Plate 5: Microstructure of Nst 60-2 Control Specimen from transverse orientation x 100 mag.

4.2 Analysis of results

4.2.1 Bridgman correction factor: Bridgman correction factor, B is the ratio of true stress to average stress and can be calculated using the expression (Mielnik, 1991):

$$B = \frac{1}{\left(1+\frac{2R}{a}\right)\ln\left(1+\frac{a}{2R}\right)} \quad \text{……… ……….(1)}$$

Where: a = radius of the neck cross section (a = 2 mm); r = the profile radius (r = 1 mm).

B = f (a, R). It is therefore the same in respect of the specimen. If $\frac{a}{R}$ = r, then B = $[(1 + \frac{2}{r}) \ln(1 + r2)]-1$. By substituting, we have:

B = $[(1 + \frac{2}{1}) \ln(1 + \frac{1}{2})]^{-1}$ = [(3) ln (1.5)] = $[3 \times 0.4055]^{-1} = [1.2164]^{-1} = 0.822$

4.3 Discussion of Results

The results showed an ultimate tensile strength (UTS) of 1768.19 N/mm^2 for NICD specimen compared with 1696.02, 1587.76 and 1551.67 N/mm^2 for CNCCD, CNCBL, and NIBL heat treatments respectively, as a result of the refinement of the primary phase after the subsequent diffusing and cooling processes. NICD heat treatment with UTS of 1768.19 N/mm^2 (Table 2) resulted in an increase of 42% compared with CNCCD, CNCBL and NIBL heat treatment with increase of 36%, 27% and 24% respectively.

CNCCD specimen records the highest Yield strength of 1299.08N/mm^2, an increase of 36% more than the controlled specimen, compared to increase of 18%, 11% and 5% for NICD, NIBL and CNCBL respectively. This comparatively shows the relative ease with which carbon and nitrogen diffuse into the surface of the specimen.

Introduction of a notch in a tensile test specimen causes yield strength to increase but ductility (% total elongation) decreased irrespective of the thermo-chemical heat treatment given to the sample (Tables 1 & 2). This increase can be explained on the basis of introduction of tri-axial tensile stresses at the root of the notch (Dieter, 1988) which causes practically zero shear stress at the notch root and hence plastic deformation is completely suppressed. Introduction of a notch in a tensile test specimen causes brittle fracture in spite of the fact that the metal is ductile. (Dieter, 1988)

From Plates 2- 5, the microstructures revealed that, hardness reduces progressively until it reaches the core hardness. This necessitated the caution for not grinding the parts excessively, otherwise the resulting surface hardness and strength will be reduced significantly. Plate 2 reveals that martensite partly transformed into ferrite (white). In Plate 3, Cementite and Carbon precipitated with martensite (black) and bainite (dark gray) region which formed needle like structure with some ferrite and cementite. Plate 4 revealed a fine-grained martensite (black), ferrite-pearlitic structure and some untransformed austenite (gray white) was formed. The control specimen, Plate 5 revealed that the steel is made up of ferrite (white) + pearlite (black). The light cementite regions are surrounded by pearlite, which has a ferrite–cementite layered structure as can be seen in the remaining plates.

4. CONCLUSION

Carbonitriding thermo-chemical heat treatments, CNCBL and CNCCD, increased the UTS of the Mild Steel by 27% and 36% respectively while Nitriding thermo-chemical heat treatments, NICD and NIBL, increased the UTS of the mild steel by 42% and 24% respectively.

Introduction of a notch in a tensile test specimen causes yield strength to increase but ductility (% total elongation) decreases irrespective of the thermo-chemical heat treatment given to the sample. Cow dung, bean leaves and wood charcoal should be processed into carburizing materials for thermochemical heat treatment.

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