INVESTIGATION OF NIGERIAN STEELS FOR CRYOGENIC APPLICATION

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ABSTRACT

Nigeria produces steels and has just entered the league of Nations which export natural gas in the form of liquified natural gas (LNG).

In order to use Nigerian steels for cryogenic temperature applications, this work is undertaken to examine the possibility of further developing the steel(s) which show promise from cryogenic temperature tests.

Samples of Nigerian popular steels, NST44-2, NST-60-2 and NST 60 Mn were machined into standard charpy impact testing specimen as specified by ASTM. These samples were heat treated by soaking for 1 hour at 930^{o}C and cooled in the air.

Impacts tests were conducted on these specimens, of as rolled and normalised conditions at room and cryogenic temperatures of 0oC (273K), -5oC (268K), -10oC (263K) and -15oC (258K).

The results show that the steel NST 44-2 is promising for cryogenic applications. The high energy absorbed accompanied by ductile fracture in NST 44-2 can be attributed to the normalising effect, where more ferrite was transformed than pearlite from ($\alpha + \gamma$) region of the heat treatment, which resulted in grain refinement and decrease in ductile-brittle transition temperatures. The ductility of the sample is due to low carbon content of the material.

INTRODUCTION

The Nigerian steel company, has been producing different grades of steels for construction and manufacturing industries for engineering component parts.

Nigeria as natural gas producing nation with abundant reserves, needs to look inwards to use her steels to manufacture the essential component parts for the gas industry. In order to use Nigerian steel for this purpose, it must be noted that the engineering properties of most structural steels change substantially as temperature is decreased to very low value, that is in cryogenic applications. Common property changes include dimensional contraction, increases in yield and tensile strengths, and decrease in fracture toughness, with the latter often occurring dramatically over a narrow temperature range near the ductile-brittle transition temperature (D.B.T.T). Austenitic steels may also undergo magnetic and structural phase transformations. The structural steels which are appropriate for cryogenic applications are hence the product of careful material selection and specific alloy design.

These cryogenic applications require structural alloy steels which combine high strength with good fracture toughness at the service temperatures and retain these properties after welding. Other materials constraints are determined by specific applications and may concern, for example coefficient of expansion, corrosion resistance, fatigue lifetime, sensitivity to nuclear radiation, magnetic properties and phase stability (1,2). Cryogenic structural steels are generally of low carbon alloys hence are required to have decrease in (DBTT), increase in fracture toughness (impact toughness) at low temperatures (3). The increased use of natural gas has greatly increased the requirements that have to be met by construction materials for storing and transport of liquid natural gas (LNG). During such handling the LNG is kept at temperatures of about –162°C, which means that a vessel containing LNG has to function at that low temperature. Obviously, strict requirements as to weldability, strength and low-temperature toughness are laid on a material for fabrication of such vessels. For safety reasons, excellent toughness, including impact properties, are required at temperatures below –162°C, and a test temperature of –196°C is therefore convenient. One material able to meet all the requirements for LNG applications even in thick plates fabricated under what production circumstances is 9% Ni Steel (4). In spite of the relatively high prices the 9% Ni steels have therefore become an important LNG material, particularly for applications in LNG tankers.

In order, to find alternative metal to nickel in cryogenic structural steels, because of the high cost, the low temperature mechanical properties of Fe-Mn alloys have been the subject of active research over the last several years (5-8). The results of these works had been promising, (9). Since nickel can be replaced by manganese in cryogenic structural steels, because the influence of manganese on the transformation behaviour of iron is similar to that of Nickel.

Also Mn and Ni are both austentic stabilising elements when added to Fe and both reduce the critical cooling speed to form martensite following an austenitising treatment. Since Nigerian steels have manganese as a minimum alloy content, it is worth while to investigate cryogenic mechanical properties, for the sake of using Nigerian steel for LNG materials, particularly for application in LNG tankers and storage facilities.

The present investigation is aimed at obtaining impact test results at some of the cryogenic temperatures on Nigerian popular structural steels. The Ductile-to-brittle transition temperature (D.B.T.T) test temperature of these type of steels lying between O°C to –20°C, according to (10). The results will form the basis for further work on the steel(s) that have cryogenic mechanical properties.

EXPERIMENTAL PROCEDURE

Three common Nigerian steels NST 44-2, NST60-2 and NST 60-Mn were used. The chemical composition of the steels is shown in Table 1. The samples were obtained from Jos Steel Rolling Company. ASTM standard specimen for the charpy (V-notched) tests, were prepared from he samples. Charpy specimens were cut longitudinal to the rolling direction and the notches were machined on the rolled surface of the plate. The specimen were heat-treated at 930°C for 1 hour, and normalised in still air. Since liquid Nitrogen is not available, for cryogenic temperatures, the following procedures were adopted. Since water freezes at 0°C, it was be difficult to generate temperatures lower than –4°C from ice blocks. However, by using the principle of freezing point depression of brine (Sodium chloride salt), temperature of –15°C (258K) were generated for the experiment.

The normalized specimens were fed into a refrigerator, whose evaporating chamber was measured to be –8°C (265K). This was to aid in lowering the temperatures of the specimen by cooling. After 15 minutes, the specimens were removed and fed into a well lagged flask containing some ice block and salt solution which was measured by thermometer to be –15°C (258K). Another 15 minutes were allowed for the specimen to attain that temperature. Then, the conventional charpy-V impact tests were carried out at room temperature and in the temperature range 0° (273K) to –15°C (258). During the tests at cryogenic temperatures, the specimens were placed on the testing machine, as quick as possible. It was ensured that not more than 5 seconds was elapsed so that specimens could remain at the test temperature.

RESULTS AND DISCUSSIONS

The impact energy absorbed by the specimens in the as-rolled and normalized condition at room temperature are shown in Table 2. As can be seen from Table 2, the impact energy absorbed by the normalized specimens of NST 44-2, NST 60-2 and NST 60 Mn showed higher values than of the as-rolled samples. The reason, of the improvements of the values of the energy absorbed was due to the heat treatment. The heat-treatment particularly in regard to the temperature, is a very important factor in obtaining the required toughness (4). The obvious reason for this is that the microstructure is a critical factor for the toughness properties. It is therefore important to optimize the micro structure, and it is wellknown that a desirable structure should contain a minimum of cementite, but instead a small amounts of very stable austenite particles in which the excess carbon content is dissolved. Such a condition in a 9% Ni steel is obtained by careful tempering (4). Several aspects of the importance of such retained austenite in enhancing the resistance against cleavage fracture in Ni steels have been given in (11-13).

It was found by Marschall et al (14) that a heat treatment within the two-phase ($\propto+\gamma$) region was effective in achieving fine grain size and retained γ in a bcc 9 Ni steel. In this work, heat treatment was in the region of $\propto+\gamma$, which could have helped to obtain fine grain size in the samples which resulted into higher energy absorptions. Bolton et al (15) studied the tempering of binary Fe-Mn alloys with varying Mn content from 4 to 10%. While the tempering treatment was shown to be beneficial to low temperature toughness no retained phase (γ or Σ) was observed in their work.

Roberts (16) showed that the mechanical properties of Fe-Mn alloys containing up to 9% Mn were strongly influenced by the grain size and the substructure of the transformation product. Schanfein (17) attempted to reduce the grain size of an Fe-8Mn alloy but was able to obtain only a minor suppression of the ductile to brittle transition temperature.

Table 3 shows the impact energy absorbed by normalized samples of NST 44-2, NST 60-2 and NST60-Mn at some of the generated cryogenic temperatures. Decrease in fracture toughness of structural steels often occur over a narrow temperature range near the ductile ransition temperature (D.B.T.T) (18). Hence, the impact energy absorbed by NST 44-2 at room temperature which was 404J, was reduced to 324J at 273K. At 268K, the impact energy was increased to 344J, then raised to 376J at 263K.

Finally as the temperature got lower to 258K, the specimen failed, with lower energy. The fractures observed in all the specimens of NST-44-2 at these cryogenic temperature were completely of the ductile type.

The energy absorbed by the normalised NST 60-2 and NST 60Mn at the cryogenic temperatures were decreased drastically and there were a fluctuation in the energy absorption during fracture. Brittle fracture was observed at all the temperatures. Looking at all the three samples of the steels, the energy absorbed by the NST 44-2 at room temperature after normalizing was higher and almost double those of NST-60-2 and NST-60-Mn. This could be attributed to more grain refinement by the heat treatment in NST 44-2 than the other two samples. Also from the knowledge of heat treatment, fine grain size with more ferrite and small amounts of pearlite might have formed in NST-44-2 due to it's low carbon content (see Table 1). This resulted in toughness and ductility which lead to the high energy absorption, with ductile failure. NST 60-2 and NST-60Mn belong to the medium carbon group with equal amounts of ferrite and pearlite are formed under normalised condition (19). Hence appreciable amounts of energy were absorbed though lower than those of NST-44-2. Recent work on NST-60-Mn by Inegbenebor (20) found out that normalized samples, have higher ductility than the quenched ones. This agrees with the higher energy absorbed in the normalized condition of the samples.

From all the results, the factor which resulted in the optimum absorbed energy in NST-44-2 can also be attributed to the decrease in the ductile-brittle transition temperature. Pickering (19) reported that the normalizing process in metals results in the decrease of the ductile brittle transition temperature.

Conclusions

NST-44-2 absorbed higher energy even at lower temperature up to 336J at 258K. It was observed also that NST-44-2 samples at various sub-zero temperatures failed with plastic deformation in the fracture region, hence with ductile fracture. This can be attributed to (i) its ductility as a low carbon steel and (ii) the decrease in the ductile transition temperature by normalizing.

On the other hand, NST-60-2 and NST 60-Mn samples both failed with brittle fracture, with decrease in energy absorbed, probably due to higher carbon content of the samples. NST-44-2 is a candidate steel for further development for cryogenic application.

Non-conclusive.

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Table 1: Chemical Compositions of the Samples

S/No.	Sample	Carbon %(C)	Manganese % (Mn)	Sulfur % (s)
1.	NST44-2	0.14-0.20	0.40-0.60	0.018-0028
2.	NST60-2)	0.35-0.42	0.60-0.80	0.018-0.028
3.	NST60Mn	0.35-0.42	0.90-1.20	0.018-0.028

Table 2: Impact Energy Absorbed at Room Temperature

S/No.	Samples	Condition of Samples	Charpy V-Impact Test Tem. °C(K)	Energy Absorbed Joules (J)
1.	NST 44-2	As-rolled	24°C(297K)	284
		Normalised After heat treatment at 930°C for 1 hour	24°C (297K)	404
2.	NST-60-2	As rolled	24°C (297K)	78
		Normalised After heat treatment at 930°C for 1 hour	24°C (297)	134
3.	NST-60.Mn	As rolled	24°C (297K)	84
		Normalised After heat treatment at 930°C for 1 hours	24°C (297K)	118

Table 3: Impact Energy Absorbed at Cryogenic Temperatures

S/No.	Samples	Condition of the Samples	Test Temp. °C (K)	Energy Absorbed Joules (J)
1.	NST-44-2	Normalised	0°C (273K)	324
		After heat treatment at 930°C for 1 hour	-5°C (268K)	344
			-10°C (263K)	376
			-15°C (258K)	336
2.	NST-6—2	Normalised	0°C (273K)	94
		After heat treatment at 930°C for 1 hour	-5°C (268K)	124
			-10°C (263K)	100
			-15°C (258K)	122
3.	N_ST-60-Mn	Normalised	0°C (273K)	
		After heat treatment at 930oC for 1 hour	-5°C (268K)	114
			-10°C (268)	96
			-15°C (258)	86