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# FATIGUE BEHAVIORA STUDY OF FERRITIC-MARTENSITIC CRYOGENICALLY TREATED USING 17Mn4 STEEL

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**ABSTRACT:** - Based on the importance of impressive mechanical properties good ductility, reduced cost, superior formability and excellent surface finish in terms of Ferritic-martensitic steel, it has been given consideration over other high strength low alloy steels and as such, this research work has been carried out modifying the properties of 17Mn4 Ferritic-martensitic steel.

In this work, the 17Mn4 ferritic-martensitic steel specimens were made and cryogenically treated as the first step towards its modification. Fatigue, micro hardness and microstructure inspections were carried out for all treated and non-cryogenic treated 17Mn4 steel specimens. The results showed a significant increase in strength of the 17Mn4 steel compared to the commercial plain carbon steels. From the conducted experiments, the results showed that the fatigue limits for the dual and cryogenic specimens been obtained as (380MPa) and (400MPa) respectively. A considerable enhancement in the fatigue value for the cryogenic steel showed a 6.25%rise as well as the hardness value for the dual and cryogenic treated specimens increasing significantly to (186) and (198) respectively. Further investigation showed a remarkable enhancement in the hardness value to be 25% for the cryo-treated specimen. While the microstructure inspection showed that, the steel containing martensitic with less ferrite grains formed around it, the investigation proved an increment in them artensitic ratio and was seen after the cryogenic treatment was conducted.

**Keywords:** Microhardness, Microstructure, Fatigue, Ferritic-martensiticSteel, Cryogenic

#### 1. INTRODUCTION

The legislation on the reduction of CO<sub>2</sub> emission through a better fuel economy has urged car manufacturers to use lighter yet stronger materials which must also meet the safety standards in terms of mechanical properties. Among the other available materials, ferritic-martensitic steel has received wider attention as this type of steel has a good combination of higher tensile strength along with significant ductility [1]. Continuous drive to provide high performance and known reliability while keeping the low overall cost of an automobile has led to an increase in the utilization of advanced high strength steels (AHSS) for the body structure. These high strength steels rapidly replace their low carbon counterparts in automotive applications in view of improved fuel efficiency through lightweight materials. In the AHSS family, ferritic-martensitic steels are used in a large scale due to their inherent high strength, good formability and low yield-to-ultimate strength ratio. Automotive components of ferritic-martensitic steels include body structures such as pillars, rails and cross members, door and inner bumper reinforcement beams and suspension housing. In these applications, this type of steel has demonstrated improved service performances in crashworthiness and

fatigue durability[2]. Fatigue is referred to the degradation of mechanical properties leading to failure of a material or component under cyclic loading as shown in figure.1 [3]. Fatigue strength of materials is often expressed by the maximum stress amplitude without failure after given number of cycles. It is obvious that, more than (90%) of service failure of metallic components might be due to the existence of fatigue. Its performance test requires time and money consuming and therefore, needs many attempts to determine the fatigue strength in economic way relating fatigue strength to other mechanical properties, such as yield strength, tensile strength and hardness [4]. The influence of microstructure on fatigue crack growth (FCG) behavior in steels has been subjected to considerable research interest for many years. Such microstructural effects on FCG are found to be most significant in the near threshold regime ( $\Delta K_{th}$ ) where growth rate of long cracks is negligible, i.e. less than 10–7 mm/cycle[5]. Evaluation of newer materials with improved combinations of strength, ductility and toughness has led to the emergence of a series of composite structures, in which ferriticmartensitic steels represent a distinguished class. The specific potentials that have been technologically exploited are their superior ductility and formability characteristics compared to advanced high strength steel (AHSS)or conventional plain carbon steels of similar strength level. Although, fatigue as a problem, affects any structural component or part that moves with the initiation considered to be a localized phenomenon that depends on the structural geometry and stress concentrations [6-8].

Cryogenic treatment is known to represents a process for the production of steel for the most different technical applications [9, 10] and its cryo-treated quality was found to strongly being influenced by the characteristics of microstructural transformations between Austenite and Martensite [11, 12]. Furthermore, cryo-treatment was therefore considered to improve wear resistance, fatigue life, and minimize residual stress [13]. While cryogenic treatment does not consider hardening of metals as a process as when compared to quenching and tempering, and as such cannot be substituted for heat-treating since it is only an addition to the heat treatment process.

However, Cryogenic quenching treatments have been accepted and adopted in commercial practice as an effective method for completing martensitic transformation in alloyed and casehardened steels [14,15]. For wear resistance improvement, the cryogenic treatments, will usually involve cooling within the temperature range of -120°C to -195°C, hence, the replacement of popular dry ice and mechanical refrigeration treatments be applied before a single or multiple tempering steps. However, reported wear resistance improvements vary between a few and a few hundred percent, wherein conflicting results are presented for the change in impact resistance of treated steels [16].

#### 2. EXPERIMENTAL WORK

# 2.1 Materials and specimen preparation

17Mn4steel according to the ASTM standard, commercially microalloyed steel has been used in the present investigation. The steel was analyzed using a spectrovac (Thermo ARL 3460, Optical Emission Spectrometer) and the chemical composition can be listed as in table 1.

The fatigue test specimens were prepared according to the (ASTME467) standard. The shape and dimensions of fatigue specimens used in this experimental work are shown in figures 1 and 2 respectively. During manufacturing of the specimens, carful control has been taken to produce good surface finish and to minimize residual stresses. The outer surface at curvature radii of all specimens was polished to eliminate the effect of surface roughness caused by machining; hence, all specimens obtained are with similar surface finish. This has been done by using different wet silicon carbide papers such that; 400, 600, 800, 1000,1200,1800,2000, and 2400µm.

In this research work, All the machined CT specimens were austenitized at 930 °C in a muffle furnace as well as they were homogenized for 60 min. The next step has been carried out by cooling them in air to get a homogenous structure. This is to ensure the same starting

microstructure in all the cases. After that, the specimens were heated to  $810\,^{\circ}$ C and then be quenched in brine water to get ferritic-martensiticsteel. Then, they were immersed in nitrogen liquid for 36hrs as a final step.

#### 2.2 Heat Treatments

The heat treatment included in this work is mainly represented by normalizing and inter critical annealing. The purpose of normalizing is to remove any effect of previous manufacturing processes, and hence it was performed on the as received steel before carrying any machining on the specimens. The procedure for normalizing treatment is carried out by heating the steel above the upper critical temperature Ac3 by 30°Cto transform the steel into fully austenitic phase, while soaking time should not be prolonged to avoid grain growth and excessive oxidation but enough to dissolve all carbides, and cooling is made in still air.

To obtain ferretic-martensitic microstructure, the steel has been subjected to inter critical annealing by heating it to a temperature region between Ac1and Ac3 at a temperature (780 °C) for (20 min) followed by quenching in brine water (15% NaCl in water). Agitation was applied during quenching to avoid the effect of vapor jacket as well.

# 2.3 Cryogenic Treatment

Liquid nitrogen has been used as cooling media to carry out the cryogenic treatment. The specimens were encased in paraffin wax to act as insulator. This enables gradual change in the temperature of the specimen to prevent any thermal shock that may occur, thus causing undesirable deformation or cracking. The process typically started by slowly cooling a mass of parts to  $-196\,^{\circ}\text{C}$ , holding the specimens at this temperature for 24 hrs. After that, they were removed and left to warm at room temperature. In case of steel, the benefits are usually attributed to the reduction or elimination of retained austenite from hardened steel and accompanied by the precipitation of small finely dispersed carbides ( $\eta$ -carbides) in the martensite. Figure3 shows a typical cryogenic treatment temperature sequence for 17Mn4 steels. However, the cryogenic treatment of the specimens has been carried out in a chamber which is fully covered with multilayer super insulation and is filled by liquid nitrogen.

# 2.4 Mechanical properties tests

#### 2.4.1 Fatigue

In this research work, rotating bending fatigue machine model (WP 140 Fatigue testing machine) has been used to carry out the fatigue test. The fatigue test for steel samples was accomplished according to the specification of (ASTM E467). This test has been achieved by using applied bending load at the end of cantilever rotating sample with fatigue testing machine speed of (2800 rpm). Furthermore, to this, the samples of the test have been prepared to final dimensions wherein the applied bending load at the end of sample was (300 N).

#### 2.4.2 Microhardness

Vickers Hardness test has been carried out by using Zwick Hardness tester equipped with a diamond square pyramid. In this test a range of loads of (200 g - 10 Kg) were used as this makes this device capable of performing micro and macro hardness tests in mm. The mean of the indentations diagonals was used to calculate the Vickers hardness numbers using the formula:

 $HV = 1.8544 P/d^2(2.1)$ 

In which, HV is the Vickers hardness, P is the applied load in Kg Force and d is the diagonals mean in mm.

#### 3. RESULTS AND DISCUSSION

#### 3.1 Fatigue test

The results of fatigue test for normalized and ferritic-martensitic steel with and without cryogenic treatment of (17Mn4) can be investigated herein . These results have been represented as (S-N) curve in figure4. According to the S-N curves, it can be seen that the maximum value of the stress (260MPa) can be obtained at a number of cycles of about (181632) for the normalized steel. Wherein, no failure can be seen for this case when the stress value is 220 MPa or less. In comparison, the value of the maximum stresses for the ferretic-martensitic steel for the cryogenic treated and no treated were found to be 400 and 380 MPa at a number of cycles 203298 and 239191 respectively. No failure can be seen for these cases when the stress value is less than 350Mpa.

Figure 5 indicates the investigated improving in the fatigue limits for the cryo-treated and non-treated dual phase steels as compared to the normalized steel. It can be seen that, the fatigue limit of ferritic-martensitic (FM) steel is in general higher than the normalized steel and this attributes to structure of FM steel which contain shard phase martensite. In fact, after the cryogenic treatment of FM steel it was observed that the fatigue limit is enhanced by a percent of (6.25). This might be due to the fact that the retained austenite transforms into martensite, or increase in the hardness of surface. Also, this can be explained due to the precipitation of transition carbide into structure of ferritic-martesitic steel because of the action of the cryogenic treatment.

As a result, the cryogenic treatment in this work found to be a strong factor that increases the high cycle fatigue strength. This Correspond with what it is reported that dual phase cryogenic treatment (DCT) has many benefits. It not only gives dimensional stability to the material, but also improves abrasive, fatigue and wear resistance as this in agree with the studies [17,18, 19,20]. Furthermore to this, the cryogenic treatment has been used to increase strength and hardness of the materials as this indicated in the studies [21-23] as well. The main reason for these improvements in properties are due to the complete transformation of retained austenite into martensite and the precipitation of ultra-fine eta- carbides dispersed into the tempered martensitic matrix [17, 24]. Numerous practical successes of cryogenic treatment and research projects have been reported worldwide [17-21, 23-28]. Several works in the literature showed that cryogenic-assisted precipitation of fine  $\mu$  carbides are related to the induced stresses arising on the martensitic structure of steels during DCT, after quenching and before tempering step.

#### 3.2Hardness test

In the present work, the Microhardness test has been performed by using 1Kg load. Where in, The lower testing load (i.e. 200 gm) found to be inappropriate for this test due to the banded nature of the microstructure of the specimen. In which the bands of carbon rich-areas will transform into martensite rich-area as this shows hardness value higher than that of the carbon poor-area which mainly formed of ferrite. To avoid fluctuation in hardness reading, both areas must be covered at the same time during the test wherein (1 Kg) load was found to be more suitable in covering both areas. Figure 6 indicates the hardness values versus the distance along the specimen surface for all considered cases. Accordingly, the microhardnesses of ferritic-martensitic steel has been measured to be (193, 178 and 193). Wherein the hardness values for the specimens, after carrying out the cryogenic treatment, were about (210, 185, and 210) in comparison to the base metal which was (159) Vickers hardness. As a result, the hardness values have increased after the cryogenic process has performed as this can be attributed to the increase in the ratio of the martensite. This has been explained due to the transformation of the retained austenite into martensite. Also, this increase might be due to the fine carbides precipitation by the effect of cryogenic treatment in the structure of ferritic-martensitic steel. Furthermore to this, table2 computes the enhancement in hardness values after carrying out the cryogenic treatment for the considered cases herein. The enhancement in the hardness values for the cryo-treated dual phase and the non-treated dual phase steel were obtained to be 24.3% and 16.7% as compared to the

normalized steel. More hardness enhancement for the cryo-treated specimen can be obtained to be 6.4 % as compared to the dual phase specimen without the cry-treating process. This change occurs because of hard phase martensite, which forms after critical annealing treatment. In addition to this, it can be noticed that the hardness increases after cryogenic treatment as result to increase volume of martensite in the structure which leads to increases the hardness as a result.

#### 3.3 Microstructure

Figure 7 shows the microstructure of normalized and the ferritic-martensitic steel before and after the cryogenic treatment. By using point counting method, it was found that the martensite content approximately reaches(23%). Wherein, the martensite content has increased to be (31%) for the steel after crying out the cryogenic treatment. This considered increase in the martensite content might be due to the transformation of retained austenite to martensite after the cryogenic treatment. Also, it can be investigated that the ferrite in ferritic-martensitic steel after cryogenic treatment contains fine distribution precipitates as this shown in figure 7. Hence, the microstructure inspection found to be in total agreement with the explanation of mechanical properties enhancement mentioned before.

#### 4- CONCLUSIONS

From the results of the present investigation, the following conclusions were drawn:

- 1- The hardness of ferritic-martensitic steel is increased after the cryogenic treatment.
- 2- Cryogenic treatment increases the Endurance limit of ferritic-martensitic steel.
- 3- ferritic-martensitic steel has a hardness value higher than that of the normalized steel.
- 4- ferritic-martensitic steel shows a fatigue limit higher than that of the normalized steel.
- 5- The microstructure inspections reveals to large amount of martensite due to the transformation of retained austenite as a result to the cryogenic treatment.

#### REFERENCES

- 1. M. Sarwar and R. Priestner, "Fatigue Crack Propagation Behavior in Dual-Phase Steel", J. Mater. Eng. Perform.8, (1999)p 245–251.
- 2. Hu ZG, Zhu P, Meng J. Fatigue properties of transformation-induced plasticity and dual-phase steels for auto-body lightweight: Experiment, modeling and application. Materials and Design 2010; 31(6):2884-2890.
- 3. Andresen, P. & Antolovich, B., Fatigue and Fracture. ASM Handbook, (1997)
- 4. Pang. J. C., Li, S. X., & Wang, Z. G., General relation between tensile strength and fatigue strength of metallic materials. Materials Science and Engineering A, 564(2013), 331-341.
- 5. Masounave J. and Bailon J.P Scr. Metall. No10 (1976)pp165
- 6. M. A. Meyers and K. k. Chawla, "Mechanical behavior of material", prentice Hall Inc, USA, (1999).
- 7. M. R. Anderson, "Fatigue crack initiation and growthin ship stractures" Technical university of Denmark, in (1998).
- 8. Newjeresy, USA, C.C. Osgood, "Fatigue Design", 2nd Edition pergamonpres, (1982).
- 9. K. Surekhaand A. Els-Botes, "Effect of Cryo treatment on Tool Wear Behavior of Bohler K390 and AISI H13 Tool Steel During Friction Stir Welding of Copper", Transactions of The Indian Institute of Metals, Vol. 65, No. 3, pp. 259-264,2012, 10.1007/s12666-012-0127-8
- 10. D.S. Nadig, S. Jacob, R. Karunanithi, R. Manjunatha, D.Subramanian, M.V.N. Prasad, GeethaSen, Abhay K. Jha, "Studies of cryotreatment on the performance of integraldiaphragm pressure transducers for space application", Cryogenics, Vol. 50, Issue 9, pp. 561–565, 2010.
- 11. K. Surekhaand A. Els-Botes, "Effect of Cryotreatment on Tool Wear Behaviour of Bohler K390 and AISI H13 Tool Steel During Friction Stir Welding of Copper", Transactions of

- The Indian Institute of Metals, Vol. 65, No. 3, pp. 259-264, , 10.1007/s12666-012-0127.2012.
- 12. N. B. Dhokey and J. V. Dandawate, "Study of Wear Stabilization in Cryoprocessed Cobalt-Based High Speed Steel", Transactions of The Indian Institute of Metals, 2012
- 13. TaylanAltan, Gracious Ngaile, Gangshu She, "Cold and Hot Forging: Fundamentals and Applications", ISBN: 0-87170-805-1.
- 14. Francois Cardarell, "Materials Handbook", 2008, ISBN: 978-1-84628-669-8.
- 15. D.L. Milam, Case-hardened stainless steel bearing component and process, U.S. Patent 5,851,313, Dec.22, 1998
- 16. G.R. Bourne et al., Effects of Cryogenic Treatment on Wear Resistance of AISI D2 Tool Steel, STLE Annual Meeting 2003, Tribology Laboratory, Univ. of Florida.
- 17. Barron, R.F. Cryogenic treatment of metals to improve wear resistance. Cryogenics 1982, 22, 409–414.
- 18. Collins, D.N. Cryogenic treatment of tool steels. Adv. Mater. Process. 1998, 23–29.
- 19. Liu, H.; Wang, J.; Yang, H.; Shen, B. Effects of cryogenic treatment next term on microstructure and abrasion resistance of CrMnB high-chromium cast iron subjected to sub-critical treatment. Materials Science and Engineering A 2008, 478, 324–328.
- 20. Johan Singh, P.; Mannan, S.L.; Jayakumar, T.; Achar, D.R.G. Fatigue life extension of notches in AISI 304L weldments using deep cryogenic treatment. Eng.
- 21. Molinari A, Pellizzari M, Gialanella S, Straffelini G, Stiasny KH. Effect of deep cryogenic treatment on the mechanical properties of tool steels. J Mater Process Technol 2001, 350–355.
- 22. Moore, K.; Collins, D.N. "Cryogenic treatment of three heat treated steels. Key Eng. Mater". 1993, 86–87, 47–54.
- 23. Bensely, A.; Venkatesh, S.; Mohan Lal, D.; Nagarajan, G., Rajadurai, A.; Junik, K, "Effect of cryogenic treatment next term on distribution of residual stress in case carburized En 353 steel". Materials Science and Engineering A 2008, 479 (5), 229–235.
- 24. Meng, F.; Tagashira, K.; Azuma, R.; Sohma, H., "Role of η-carbide precipitation in the wear resistance improvements of Fe-12Cr-Mo-V-1.4C tool steel by cryogenic treatment". ISIJ Int. 1994, 34, 205–210.
- 25. Mohan Lal D, Renganarayanan S, Kalanidhi A., "Cryogenic treatment to augment wear resistance of tool and die steels cryogenics", 2001;41:149-155.
- 26. Lewskovsek, V.; Kalin, M.; Vizintin, J., "Influence of deep- cryogenic treatment on wear resistance of vacuum heat-treated HSS". Vacuum 2006, 80, 507–518.
- 27. Popandopulo, N.; Zhukova, L.T., "Transformation in high speed steels during cold treatment. Met. Sci. Heat Treatment", 1980, 22 (10), 708–710.
- 28. Firouzdor, V.; Nejati, E., Khomamizadeh, F., "Effect of deep cryogenic treatment next term on wear resistance and tool life of M2 HSS drill". J. Mater. Process. Technol. 2008, 206, 467–472.

Table 1 Shows the Chemical composition for the steel used	Table 1	Shows the	Chemical c	composition	for the	steel used
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Element (%) Steel	С	Si	Mn	P	S	Cr	Мо	Ni	
C20D	0.16	0.25	0.78	0.03	0.006	0.223	0.024	0.117	0.277
Standard	0.14-0.21	0.10-0.40	0.50-0.80	Max 0.04	Max0.04	Max 0.25	Max 0.025	Max 0.12	Max 0.3

Table 2 shows hardness values enhancement after cryogenic treatment of ferritic-martensitic steel.

		Increase or decrease %		
Type of treatment	Average of the reading	Compared with	Compared with (DP)	
		normalizing		
Normalizing	159			
DP	185.5	+16.7		
DP + CT	197.5	+24.3	+6.4	



Fig.1 photograph of fatigue specimens

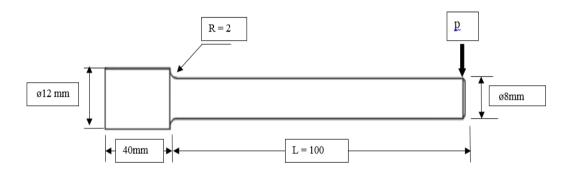


Fig.2 Fatigue test sample dimensions

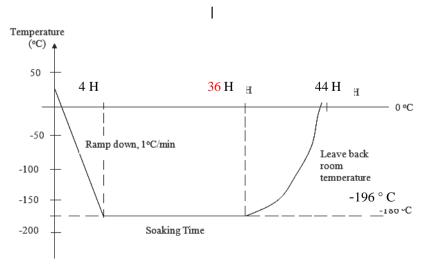


Fig.3 a typical cryogenic treatment for 17Mn4 steels.

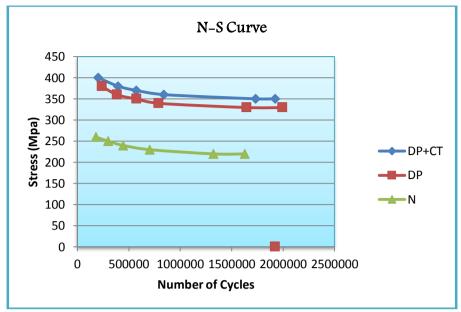


Fig.4 fatigue limits before and after cryogenic treatment

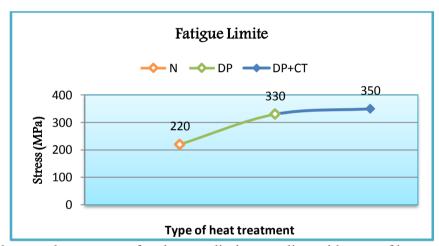


Fig.5 shows enhancement of endurance limit according with type of heat treatment

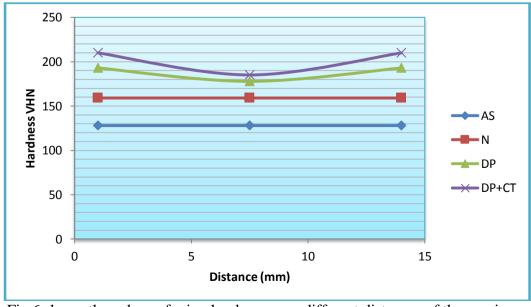


Fig.6 shows the values of microhardness over different distances of the specimen

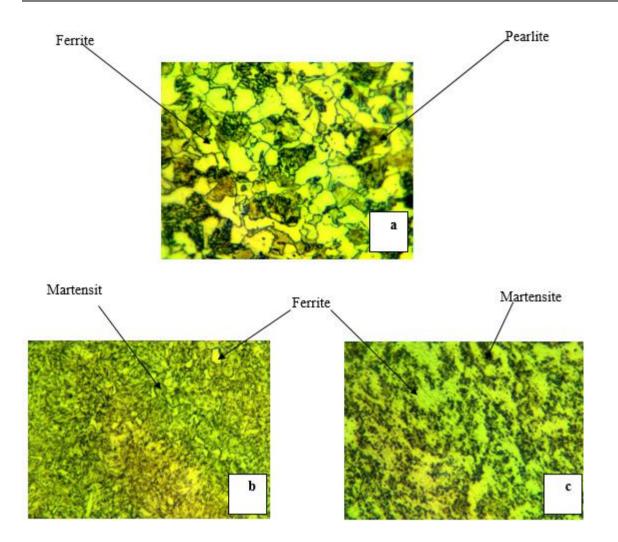


Figure 7 shows the microstructure of  ${\bf a}$ -normalized steel  ${\bf b}$ -ferritic-martensitic steel before  ${\bf c}$ -after cryogenic treatment.