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The Effect of Boriding Treatment on the Fatigue Resistance of X70 Steel

Halima Khelifi[1*], Ahmed Daas[2], Sami Zidelmel[1], Omar Allaoui[1]

[1] Process Engineering Laboratory, University of Laghouat, Laghouat 03000, Algeria
[2] Faculty of Science and Technology, Department of Mechanical Engineering, University of Djelfa City, Djelfa 17000, Algeria

Corresponding Author Email: halima.khelifi@lagh-univ.dz

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ABSTRACT

The boriding treatment is a very effective technique for hardening the surface to improve resistance to wear and friction, but it can also have negative effects on the mechanical properties and fatigue resistance of steel treated with this hardening technique. Boride parameters, such as temperature and holding time, affect the microstructure of the base metal and the boride layer. This study is interesting and important because understanding the effect of boriding and boriding parameters on the fatigue strength of X70 steel may have significant implications for industrial applications where fatigue is a major concern, such as offshore structures and transport equipment components. In order to better understand the effects of the boriding process on fatigue resistance, we carried out a series of fatigue tests on samples of X70 steel that had undergone different treatments. Three treatments were carried out, such as boriding treatment alone; direct quenching alone, and boriding followed by direct cooling. After analyzing the test results, we can evaluate the effect of each treatment on the fatigue properties of X70 steel and determine the optimal treatment combination to improve service performance. The results showed that boriding treatment followed by direct quenching has higher fatigue resistance than boriding treatment alone. Experimental data from microhardness tests showed that direct quenching after boriding increased the hardness of the base metal after boriding, which made it possible to obtain a balance between the surface hardness and the hardness of the interior of the metal. The morphology, microstructure, thickness of the borided layer, and crack origin sites were determined by means of the scanning electron microscope (SEM), an optical microscope, and microhardness by a microhardness tester.

1. INTRODUCTION

The increasing needs for materials with high properties, those working in the field of materials engineering must improve the surface properties of metals, and the subject of this research is essential to meet the increasing requirements for industrial applications. Among the various techniques for hardening the surface of material surfaces, boriding technique stands out as an effective technology to increase the surface hardness, wear resistance, and corrosion resistance of X70 steel [1, 2]. Our objective is to study the effect of boriding treatment on X70 steel, due to its excellent mechanical properties. We studied the effect of boriding parameters, such as temperature and holding time, on the microstructure and mechanical properties of steel, as well as their effects on fatigue resistance [3]. This study is considered a modern study if we take into account the materials used and the processing methods and techniques to which the samples were exposed in order to obtain results that clarify the relationship between the negative impacts of thermochemical treatment. We specifically conducted this study to achieve high hardness on the metal surface of x70 steel, and one of the goals we want to achieve is to study the effect of processing conditions on the microstructure and mechanical properties because the conditions for performing the processing are at a high temperature and for a long time, which has a direct impact on its strength. Fatigue strength of steel x70. It was expected that transformations would occur during processing, as large stresses would be generated between the surface of the sample and the borided layer, an increase in grain size, the precipitation of some metallic carbides, and the formation of a new microstructure with weak mechanical properties compared to the initial properties. This is what prompted us to search for possible and effective solutions to this problem, and one of the solutions that we used after thermochemical treatment is heat treatments. We chose the direct water quenching heat treatment after the boriding process due to the decrease in the metal core's hardness and the significant discrepancy between the borated surface's hardness and the substrate core's hardness. This is due to the negative effects of high temperatures and the prolonged boriding treatment, which resulted in a change in the metal's microstructure [4]. The grains became larger, leading to a significant change in mechanical properties such as hardness and fatigue resistance

[5, 6]. The purpose of all this is to create a hardness gradient between the surface hardness and the interior hardness of the substrates. (Quenching treatment is correction treatment.) By performing heat treatments before and after drilling processing, we performed a series of micro-hardness measurements, a set of rotary bending tests for all samples, and then analyzed the results to determine the SN (number of stress cycles) curve. We then compared the results of all samples to evaluate the effect of different treatments on fatigue resistance. We seek to establish a link between boriding and quenching heat treatment so that we can evaluate in depth the effect of quenching treatment after boriding on the fatigue resistance of X70 steel. We also seek to find a link between the boriding treatment and the fatigue performance of X70 steel, thus offering perspectives for the optimization of surface treatments in critical applications [7].

The results of our study revealed valuable observations regarding the effect of surface treatments on the microhardness of X70 steel. We noticed that there was a decrease in the microhardness value resulting from the effect of the boriding treatment; the measured value is 150 HV, compared to the hardness value of the untreated substrate of 220 HV. It was observed that there was a significant increase in microhardness when the boriding treatment was followed by the direct quenching treatment. The first result of this study showed that hardening by dredging treatments had no beneficial effect on the fatigue strength of X70 steel. On the other hand, the direct quenching treatment that followed the boriding treatment gave a beneficial effect on fatigue resistance. The results of the fatigue tests demonstrated that the specimens which underwent the boriding treatment followed by direct quenching increased the fatigue life by approximately 75%. Boride treatment also reduced sample life by approximately 62%, while direct quenching treatment increased fatigue strength by approximately 10%. These results are compared to control samples.

Therefore, combining boriding and quenching treatments may be one of the best treatments to increase the fatigue life of X70 steel whose surface has been hardened by boriding technique [8, 9]. Through a series of rotary bending tests and microhardness measurements, we seek to establish a link between the boriding treatment and the fatigue performance of X70 steel, thus offering perspectives for the optimization of surface treatments in critical applications.

In summary, control of processing parameters is essential to avoid excessive internal stresses due to differential shrinkage between the surface layer and the core of the part, and the results show that the combination of etching and quenching provides a robust solution for parts that require surfaces with high hardness while maintaining good overall flexibility. This method is especially useful for tools, gears, and other important components in the mechanical and manufacturing industries.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

Table 1 presents the Chemical analysis of the X70 steel used in this study and it shows that it meets the required specifications.

The main elements are present in the appropriate proportions, ensuring that the steel has the necessary properties for its intended use. This compliance ensures the reliability and quality of the material for the intended applications. Table 2 presents the most important mechanical properties of the steel used in our work.

Table 1. Chemical composition of X70 steel (wt.%)

Fe	C	Mn	Si	S
Balance	0.07	1.51	0.33	0.001
P	**Nb**	**V**	**Ti**	**Al**
0.012	0.046	0.049	0.003	0.035

Table 2. Mechanical properties of X70

R_m (MPa)	R_e (MPa)	A%	Microhardness
610	490	22	220 HV

2.2 Boriding treatment

In this study, a solid-voice boriding test was conducted using a powder mixture consisting of three components: a boron source, an activator and a diluent as detailed in Table 3. The boriding treatment was conducted at a processing temperature of 950°C, with a holding time of 4 hours in a muffle furnace, under standard operating conditions.

Table 3. Proportion of boriding powders

B_4C	$NaBF_4$	SiC
5%	5%	90%

2.3 Direct Quenching after Boriding

Heating process in a neutral molten salt bath for the borided test sample

- Preparation of Borided Test sample: The borided test sample are carefully cleaned to remove any surface impurities before immersion in the neutral molten salt bath. This preparation is essential to ensure optimal interaction with the molten salts.

- Immersion in the molten salt bath (initial heating):

The borided test sample are placed in a stainless-steel mesh basket with 4 mesh and 1/16″ wire diameter (soaking basket) before being immersed in a bath of neutral molten salts at an intermediate temperature of 800°C for 30 min, to allow partial transformation. This intermediate heating helps reduce internal tensions and improve ductility without compromising the hardness obtained during initial quenching.

- Immersion in water:

Before you begin to remove the test pieces of neutral molten salt, it is necessary to take safety precautions. Wear protective glasses and gloves to protect eyes and hands. Make sure the tweezers used are clean, dry and free of any residue. This will avoid any potential risk when the tongs come into contact with the molten salts. After treatment in the molten salt bath, the specimens are removed and cooled in water to room temperature in a controlled manner.

2.4 Quenching treatment

After heating the borided and the base material samples to a temperature of 800°C for 30 minutes, they were subjected to direct quenching (DQ) by immediate immersion in water at room temperature. This process aimed to achieve a two-phase structure composed of ferrite and martensitic phases. The resulting structure and morphology differ significantly from the initial state. Table 4 presents the initial information concerning the experimental conditions applied in this work during each treatment (type of treatment, temperature, holding time).

Table 4. Type of treatment and condition

Type of Treatment	Condition
Boriding	950℃ for 4 h
Direct quenching	800℃ for 30 min
Boriding + DQ	950℃ for 4h + 800℃ for 30 min
No treated	-----

2.5 Microhardness tests

Hardness tests were carried out on three types of samples: those having undergone a boriding treatment, those having undergone a heat treatment after boriding, and the untreated raw steel samples. These measurements were carried out on the surface of a polished section, using a load of 100 g with a MITUTOYO model MVK-H2 hardness tester. The objective of these tests was to evaluate the hardness of the surface layer formed by the boriding treatment, as well as to analyze the effect of temperature and treatment duration on the internal hardness of the metal. After obtaining the results, a comparison was carried out to determine the importance of the heat treatment applied after the boriding process, in terms of improving the mechanical properties of the X70 steel

2.6 Rotating bending fatigue tests

In the longitudinal direction, the parts were cut from a 14 mm thick X70 steel sheet and then machined to obtain cylindrical samples. According to the ISO, code 1143 standards (Gabriela, 2019).

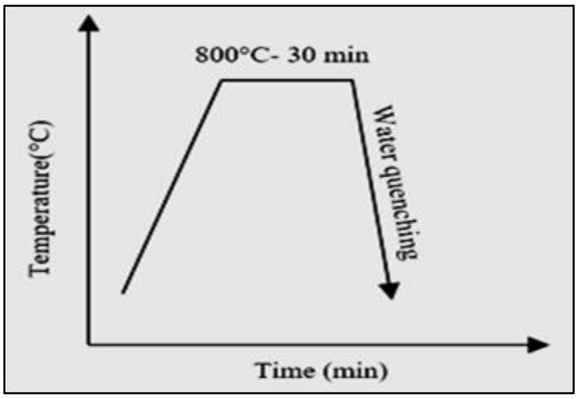

Figure 1. Diagram of heat treatment process

Figure 1 shows the dimensions and geometry of rotary bending fatigue test samples. For the rotating bending tests, four groups of fatigue specimens were prepared, each including 08 samples. The first set of samples was left without heat treatments, to serve as a reference for evaluating subsequent treatments. The second group includes samples that were subjected to boride treatment with a boride layer with a thickness of 220 um, with the aim of studying the effect of the boride layer and boriding standards on the fatigue resistance of X70 steel. The third group consists of samples quenched in water at a temperature of 800℃. In order to examine the effects of the direct quenching on the mechanical properties. Finally, the fourth group includes borided samples followed by a direct water quenching treatment, making it possible to analyse the combined influence of boriding and quenching on fatigue resistance. These groups of samples are essential for a thorough comparison of different processing methods and their effects on the fatigue performance of steel. The specimens were tested in rotary bending where the fatigue behaviour was studied at a loading frequency of 30 Hz and a loading ratio R = −1, in air, at room temperature.

3. RESULTS AND DISCUSSIONS

3.1 Microstructure

The results obtained showed that a boride layer was produced on the substrate interface of the X70 treated at 950℃ for 4 hours. It should be noted that the boride layers formed on the surface consist of Fe_2B borides. The morphology of the needle-toothed saw is characteristic of all boride layers formed on the surface of X70 steel. The thickness of the boride layers estimated is of the order of 220μm for Fe_2B. In Figure 2, we can easily observe the borated layer obtained with its acicular shape and the layer is type (D).

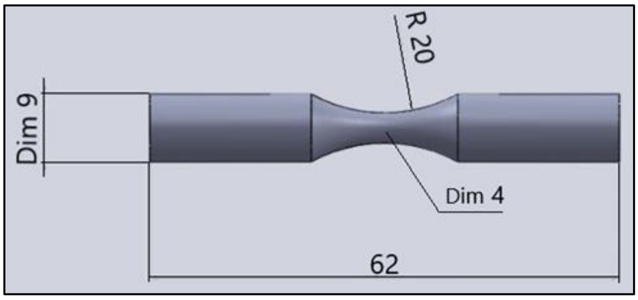

Figure 2. Rotating bending fatigue testing sample

3.2 The direct quenching effect

Microstructure analysis of API X70 steel after direct quenching treatment was carried out using an optical microscope as shown in the Figures 3 and 4.

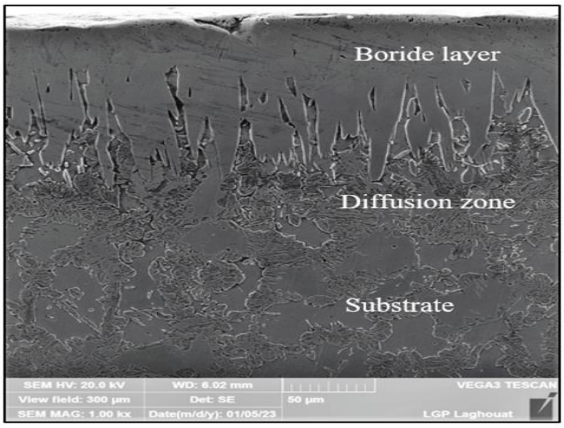

Figure 3. Borided substrate followed by a direct quenching treatment at 800℃ 30 min

This heat treatment was carried out at a temperature of 800℃ with a holding time of 30 minutes. Microscopic observations reveal that the resulting microstructure is composed of coaxial grains of ferrite, surrounded by islands of martensite. This structural configuration is significant because it illustrates the direct impact of heat treatment on the distribution and morphology of phases in X70 steel [10, 11]. Ferrite grains, known for their ductility and good toughness, contribute to the overall resilience of the steel, while martensite islands, which are harder and more resistant, reinforce the mechanical strength of the steel. This coexistence of ferrite and martensite in the microstructure thus optimizes the mechanical properties of the steel, offering a balance between strength and ductility. The presence and distribution of ferrite and martensite phases are crucial in determining the fatigue properties of steel, since martensite can introduce internal stresses that influence fatigue resistance [12, 13].

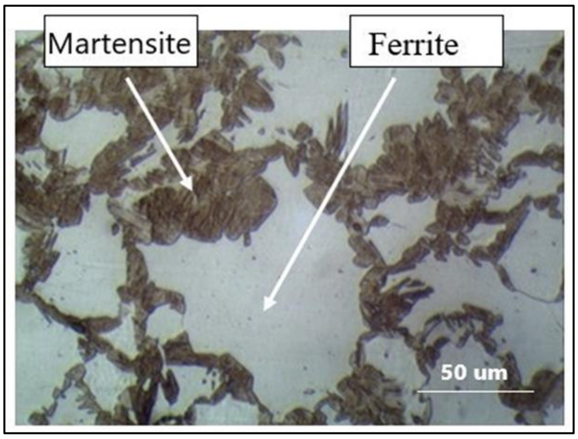

Figure 4. Ferritic-Martensitic microstructure of X70 steel after boriding treatment

3.3 Microhardness

The results obtained show a remarkable decrease in microhardness for substrates having undergone a boriding treatment. This decrease is attributed to the combined effect of the high temperature and the prolonged holding time during the boriding process. The microhardness value recorded for these substrates is 160 HV, which represents a significant decrease compared to the initial microhardness of the material, which is 230 HV. This decrease in hardness it related to changes in the microstructure of the metal, such as grain growth. It is essential to note that the temperature and duration of the boriding treatment must be controlled to minimize this adverse effect. Whereas, the substrates direct quenched after boriding showed a significant increase in microhardness, reaching up to 400 HV. This improvement is attributable to the martensitic transformation that occurs during quenching, as well as the formation of precipitates in the matrix, such as vanadium and niobium carbides. These precipitates help to strengthen the matrix and increase the hardness of the substrate. This increase in microhardness after quenching indicates that this heat treatment after boriding is effective in recovering and reinforcing the hardness of the substrate. Figure 5 and Table 4 present the variation in the microhardness values of the X70 steel substrate before and after different treatments.

Table 4. Microhardness of internal zone of X70 steel

Groups of Specimens	Microhardness
Borided	160 (HV)
Base material	220 (HV)
Direct quenching	310 (HV)
Borided +Direct Quenching	400 (HV)

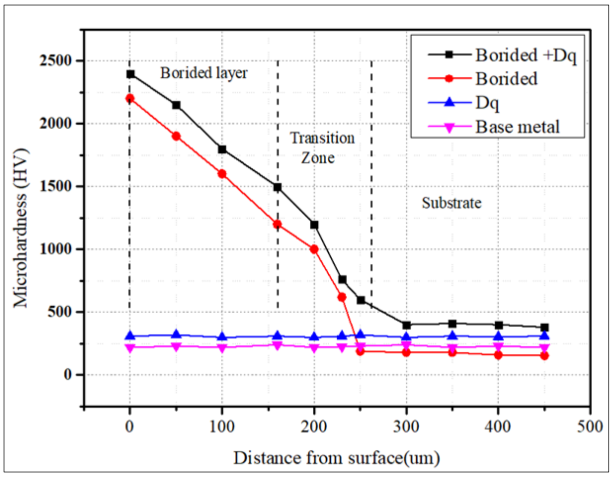

Figure 5. Surface Microhardness profiles for X70 after and before treatment

This study demonstrates that boriding treatment significantly improves the hardness and wear resistance of X70 steel, but emphasizes the need to consider its impact on the metal's microstructure. Combining boriding with direct quenching offers a promising approach to achieve optimal mechanical properties. Thus, providing a balance between the external and internal hardness of the sample treated.

The hardness values obtained in this study are consistent with the hardness value given in the literature. The micro hardness value of the boride layer FeB is 2400 HV and 2200 HV for boride Fe_2B for (B + Dq), a value of 2300 HV for FeB and 2000 HV for Fe_2B these values concern only borided substrates as well as on the underlying areas the hardness is 750 HV [14].

3.4 Fatigue test

To evaluate the influence of boriding treatment on fatigue performance, S-N curves were generated for various X70 steel specimens subjected to different treatments. These included specimens with boriding treatment only, specimens quenched after boriding, specimens directly quenched, and untreated control specimens. The testing regime covered a range of 10^4 to 10^7 cycles under rotating bending stress at 30 Hz. As detailed in Figure 6 and Table 5, a significant variation in fatigue life was observed between the treatment groups.

Table 5. Rotating bending fatigue strength

Type of Treatment	(10^4) Cycles	(10^5) Cycles
Boriding	310 MPa	240 MPa
Dq after Boriding	450 MPa	37 0MPa
Base materiel	480 MPa	390 MPa
Direct quenching	500 MPa	430 MPa

The Specimens treated with direct quenching after boriding treatment exhibited a 79% increase in fatigue life compared to those with boriding treatments only [15], highlighting the effectiveness of this combined approach in enhancing the material's durability under cyclic loading conditions. The specimens that underwent direct quenching exhibited a significant increase in fatigue life, with an approximate 79% improvement over the borided specimens and a 10 % increase compared to the control specimens. The boriding treatment decreased the fatigue life by approximately 62% compared to that of the control specimens. It has been observed that boriding negatively affects the fatigue resistance of steels, particularly when the thickness of the boride layers ranges between 100 and 350 µm, this observation was confirmed after analyzing the results of several research studies [16]. The fatigue limit σ_e was determined at the number of cycles N =10^5 using a staircase procedure. The fatigue limit σ_e in the case of the borided substrate was σ_e = 240 MPa and for the specimens having direct quenching after boriding treatment σ_e = 370 MPa the increase is 54%, as show in Figure 6 and Table 6. Fatigue rate σ_e/R_m went from σ_e /R_e = 0.48 to σ_e/Re= 0.75. In the case of the untreated control specimens, the fatigue limit σ_e was σ_e = 390 MPa and for the specimens having direct quenching only σ_e= 430 MPa, the increase is 10%, as shown in Figure 6 and Table 6. The fatigue ratio σ_e/R_e has increased from σ_e /Re = 0.78 to σ_e/Re = 0.87. It is evident that direct quenching itself can also increase the fatigue limit of the material, although the increase is not as great as that observed in the boriding process followed by direct quenching.

Table 6. Fatigue rate at 10^5 cycles

Type of Treatment	Fatigue Rate	
	σ_e/R_m	σ_e/R_e
Control sample	0.63%	0.78%
Borided sample	0.39%	0.48%
Direct quenching	0.70%	0.87%
Borided + quenching	0.60%	0.75%

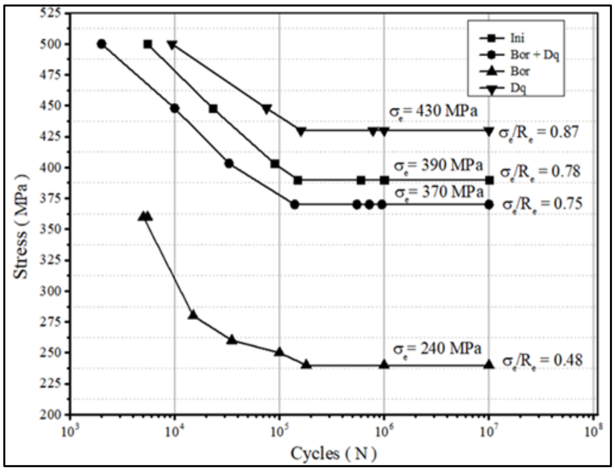

Figure 6. S–N curves of X70 steel before and after deferent treatments

The analysis demonstrates that treatments like direct quenching following boriding substantially enhance the fatigue resistance of materials by beneficially altering their microstructure. This improvement increases the material's ability to endure repeated stress cycles, this is essential for applications that require a material with high resistance to fatigue and wear. The results emphasize the importance of continued research to optimize the parameters of the boriding treatment to effectively enhance the fatigue resistance of X70 steel.

3.5 Fractographic Analysis

For samples that have been subjected to boriding treatment, it has been observed that cracks become visible to the naked eye and are confirmed by SEM Figures 7 and 8.

This phenomenon highlights the possibility of the treated surface to develop structural discontinuities under the effect of repeated stresses in the cases of thick layers with the presence of vertical cracks which propagate vertically inside the base metal and concentred residual stresses in the substrate surface due to differences in the expansion coefficients thermal [17]. This observation shows us the potential weaknesses in thick boride layers, which are thicker than 25 microns, where cracks initiate and propagate. Examination of the fracture surfaces using a scanning electron microscope (SEM) Figure 8 shows that rupture of hollow specimens is the result of crack propagation initiated at the substrate surface shape.

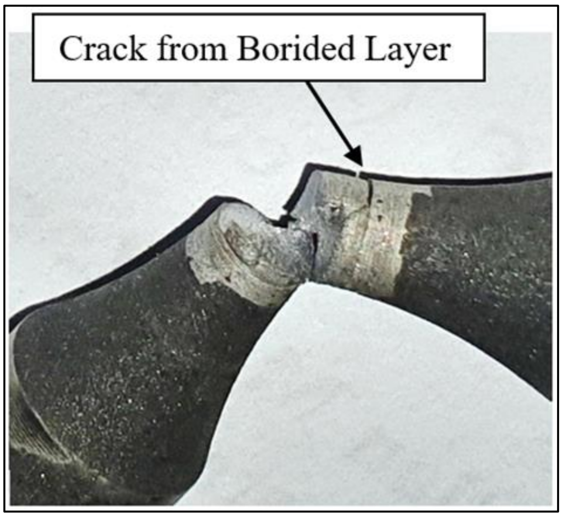

Figure 7. The fractured surface of borided substrate after fatigue test

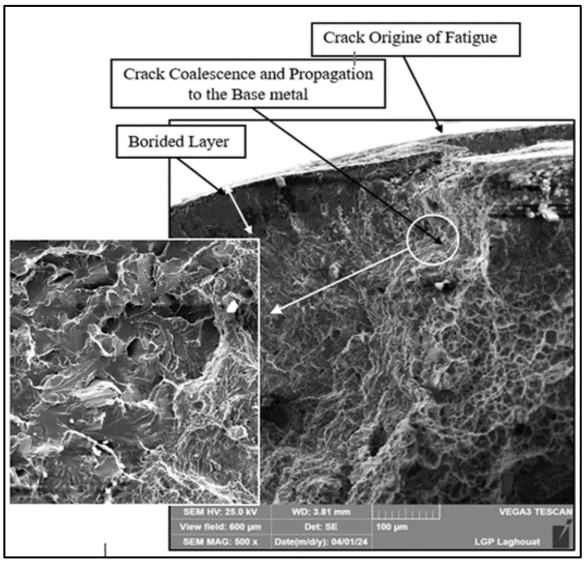

Figure 8. Fractured surface from borided substrate after rotating bending fatigue test

Figure 9 shows the initiation and propagation of multiple cracks from the base metal surface under the influence of a stress level of 400 MPa. SEM analysis of the sample reveals the presence of dimples, indicating a rapid fracture zone. We observed crack propagation trends in different surface areas. We observed crack propagation trends in different surface areas. This examination also provided us with valuable detailed insight into the fracture behavior of the material during different stress conditions, and emphasizes the importance of surface defects and stress concentrations in crack initiation and propagation.

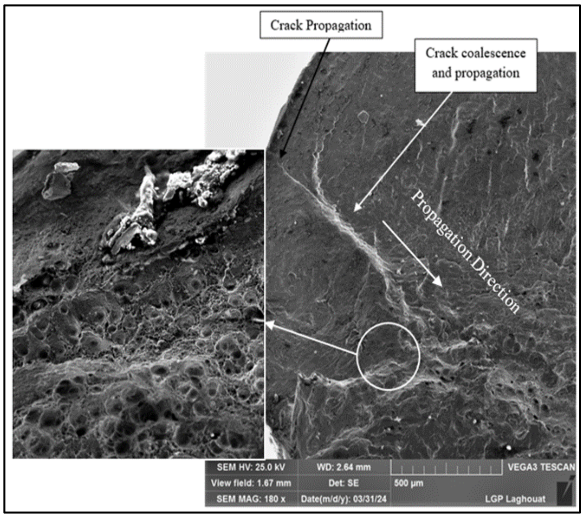

Figure 9. Fractured surface from base metal after rotating bending fatigue test

Figure 10 shows the typical crack surface of specimens subjected to direct cooling under a pressure of 430 MPa for 10^6 cycles. We notice the presence of a smooth, elongated region on the fracture surface, known as the fatigue mirror, which resulted during rubbing of the crack surfaces during cyclic loading. The presence of this mirror-like region is an indication that the component held up for a longer period before it underwent complete failure. When a crack propagates in the material under cyclic loading, the crack surfaces can come into contact and undergo relative motion, resulting in smoothing of the fracture surfaces. We also observed a zone of crack coalescence and propagation, followed by a zone of rapid cracking.

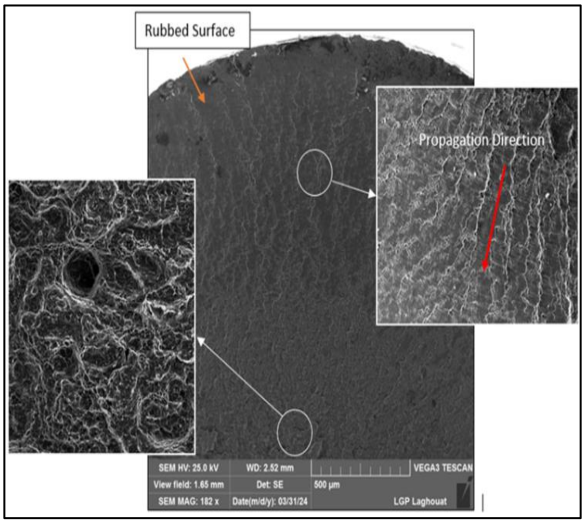

Figure 10. Fractured surface from direct quenching substrate after rotating bending fatigue test

4. COMPARISON WITH OTHER STUDIES

We mentioned previously that this work is recent and has not been studied before, but we found a study that was carried out on the same metal and where the process of mechanical hardening treatment of the surface of the specimen will undergo a rotary bending fatigue test to determine fatigue resistance. The aim of the study was to create surface stress on the metal, in order to increase fatigue resistance. We will present and compare the results obtained. For both studies and obtain a conclusion.

Shot peening application causes deformation strengthening of surface layers of tested steel, fatigue limit determined for smooth as machined specimens was σ_e = 380 MPa and for smooth shot peened specimens σ_e = 400 MPa, the increase is 5.26 %. The fatigue ratio σ_e / Rm increased from σ_e / Rm = 0.628 to σ_e / Rm = 0.661 [18-20].

Through the comparison Table 7, we notice that the heat treatment process had a good effect, as we recorded a value of σe = 430 MPa, while a value of σe = 400 MPa was recorded for the mechanical treatment of the surface.

Table 7. σe stress values comparison

Type of Treatment	Our Study σe (MPa)	Other Study σe (MPa)
Control sample	390	380
Shot peening	-	400
Borided sample	240	-
Borided + quenching	370	-
Direct quenching	430	-

5. CONCLUSION

The boriding treatment has great benefits in terms of improving wear and corrosion resistance. It also has negative effects on microstructure and mechanical properties. We need to explore these effects further. The study of the effects of boriding, direct quenching in the intercortical domain and boriding followed by direct quenching on the fatigue behavior of X70 steel. The conclusions that we can draw after the SEM and metallographic analyses as well as the rotating bending fatigue tests and the microhardness measurements were performed in order to evaluate the effects of these treatments on X70 steel. The first result of this study showed that the hardening treatments did not have any beneficial effect on the fatigue resistance and hardness of X70 steel. On the other hand, the direct quenching treatment that followed the boriding treatment had a beneficial effect on fatigue resistance. The test results showed that there was a significant increase in the fatigue life of the samples by approximately 79% compared to the results of treatment with boriding only. The boriding treatment reduced the fatigue life of the samples by about 62%, while the direct cooling treatment led to an increase in the fatigue resistance of the samples by about 10% compared to the controlled specimens. These results are in comparison with the controlled specimens. Consequently, a direct quenching treatment may be one of the best treatments to increase the fatigue life of X70 steel, whose surface has been hardened by boriding treatment at 950°C for 4 h. It should be noted that the combination of boriding and direct quenching creates an optimal balance between the surface hardness and internal hardness of the metal, indicating an effect that can be beneficial in extending the life of the treated components.

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