

Improvement of Surface Properties of 30CrMo12 Steel Using Borocarbonitriding Treatment

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Abstract - Thermochemical treatments of low alloyed steels play an essential role in improving the service life of parts under harsh conditions. Often, the treatment uses require technical significant and financial resources. For this reason, the thermochemical treatments in traditional ovens deserve consideration. In present paper, an experimental study on a 30CrMo12 steel uses the technique of borocarbonitriding process, based on the determination of the borocarbonitrided thickness layers and the kinetic constants of diffusion carried out. This nuance is used in manufacturing mechanical parts specifically solicited in fatigue as the transmission gearings in the helicopters' rotors and the rolling of in aeronautics. Borocarbonitride treatments were performed in the temperature range of 520°C for 8h. The Borocarbonitride modified surface layer samples consist mainly of γ' , ε , α , FeB, Fe2B, and Fe3C phases; according to metallographic technique analysis, it seems to be essentially a modification of the austenite matrix. High hardness values are observed in the modified layer with a sharp decrease in matrix values.

Keywords : Borocarbonitriding, 30CrMo12 Steel, Microstructure and Microhardness.

I. INTRODUCTION

The requirement to extend the life of mechanical parts at a The requirement to extend the life of mechanical parts at a
treduced cost prompted the development of surface treatments. While, surface treatments make it possible to condition a mechanical part as economically as possible for service stresses [1]. They are several; some are carried out without adding new chemical elements and the others improve the base metal by adding new element cases of all thermochemical treatments [2]. For effective surface treatment, present choice fell on the borocarbonitriding technique. Borocarbonitriding [3-15] is a thermochemical treatment typically used to diffuse nitrogen [16] carbon [17] and boron [18] into ferrous metals. This treatment plays a major role in modern manufacturing technologies to improve mechanicals, wear, and corrosion resistance of steel. There are several studies about these improvements in different types of steels can be found in the literature [19-22]. Additionally, according to the treatment process parameters; time and temperature, the alloying elements affect the formation, thickness, and hardness of the borocarbonitride layer. Borocarbonitriding has a several advantages such as a lower cost and does not required sophisticated equipment or labor skilled compared to conventional techniques, and also has a wide application in industry.

The aim of present study is investigated the microstructure and the microhardness of 30CrMo12 low alloyed steel treated by the borocarbonitride process based on the determination of the thickness of borocarbonitride layers and the kinetic diffusion constants. The used technique is carried out by analyzing images and microhardness profiles (Hv0.2). The first part allowed estimating of the borocarbonitride layer depth, while the second, in the other side, made it possible to delimit the borocarbonitride layer and deduce its depth. Scanning Electron Microscope (SEM) observations were used to highlight the effects of the elements of C, N, and B interactions in forming of the intermetallic phases, which confirmed by X-ray diffraction. The results showed an improvement of corrosion resistance and increasing in values of microhardness for the surface test pieces.

II. Materials and Method

A series of experiments were carried out to investigate the borocarbonitriding of low alloy steel 30CrMo12. The chemical composition of 30CrMo12 was analyzed by Energy Dispersive Spectroscopy (EDAX) as shown in Table 1.

Table 1. Alloying elements of steel 30CrMo12 (wt.%)

A preliminary treatment was carried out on the different samples:

- Austenitization inside furnace with a heating salt bath of composition $NaCl + KCL + BaCl2$, at a temperature of 1200°C.
- Under controlled atmosphere, an income was carried out in a furnace.

This borocarbonitriding thermochemical treatment uses a powder with a chemical composition based on carbide and nitride boron. The samples covered by a paste layer and placed in a hermetic enclosure as illustrate in Fig. 1 to prevent gases from escaping during process. A relatively thick layer of sand is added to the cover to limit air infiltration to ensure a good seal. Then, the group

is set up into an electric oven (Fig. 2) at a temperature of 520°C for 8 hours, followed by slow cooling, and then the parts are cleaned. The borocarbonitriding parameters were constant as a previous work.

Surface morphology was realized for the samples by optical microscopy and SEM. On the other hand, X-ray diffraction was performed with Co Kα radiations to determine their structure.

The chemical composition of the borocarbonitrided layers was verified by EDAX. While, the test pieces were polished for preparing in microscopic observation, and electrochemically etched with a Nital composition (3% nitric acid, 97% ethyl alcohol) to highlight matrix structure and borocarbonitride depth. X-ray diffraction analyses were obtained using Co Kα tube in Bragg-Brentano geometry in the range of 20° to 80°. Finally, the microhardness were realized using OPL to confirm the layer thickness and to evaluate its uniformity.

Fig. 1. Borocarbonitriding set up **Fig. 2.** Electric furnace

III. RESULTS

A. Macroscopic observation

Fig. 3 and Fig. 4 illustrate optical microscope and SEM observation respectively; which indicate that after borocarbonitriding, the microstructure shows multilayer form. The borocarbonitriding treatment at 520°C and 8h produced different borocarbonitrided layers in terms of morphology, thickness and phase structure [23,18, 8]: α phase (nitroferrite), corresponding to the steel matrix, ε-Fe2-3N phase of compact hexagonal structure, with a nitrogen mass fraction of 11% , γ' -Fe₄N phase of face-centered cubic structure, containing 5.80 % of N, FeB phase richer in boron $(16.23\%$ by mass), the Fe₂B phase containing 8.83% by mass of boron where the interfaces of the layers of iron borides are flat (L-type). It also forms chromium nitrides CrN $[24]$ and chromium borides (CrB and Cr₂B) $[18]$.

In the diffusion layer, one can note the presence of iron carbides attributed to the precipitation of Fe3C, generally occurring at the grain boundaries. Before borocarbonitriding presents the structure of chromium carbides $Cr_{23}C_6$ [25]. These carbides are transformed into nitrides by the arrival of the flow of nitrogen atoms; the carbon, thus released, diffuses towards the non-borocarbonitrided core and precipitates in the form of cementite Fe3C with a very low carbon mass fraction under the surface, which gradually increases until it reaches the initial value (0.3%C) relatively at 200μm from the surface, and exceeds it to the nonborocarbonitrided core. This translates well to this back diffusion

of carbon under the surface, adding the decarburization due to hydrogen atoms in the atmosphere.

The micrographic observation by SEM of borocarbonitrided sample at 520°C during 8h (Fig. 4) shows the formation of an outer layer which increases during the process to achieve a thickness about 20μm while the intermediate layer is in average of 175μm which can see a mass fraction gradient of nitrogen, carbon and boron as mentioned previously.

Fig. 3. Optical micrograph of borocarbonitrided steel 30CrMo12 at 520°C during 8 h

Fig. 4. Micrographic by SEM of borocarbonitrided sample at 520°C during 8h

B. X-ray Diffraction

X-ray diffraction of the borocarbonitrided steel 30CrMo12 at 520°C during 8h has made it possible to have the diffraction spectra study, which makes it conceivable to identify the phases present in the foreseeable borocarbonitrided layer from the balance diagram.

XRD pattern as shown in Fig. 5 observed that the treated sample consists of a mixed structure of ε-Fe₂₋₃N, γ'-Fe₄N, α phase, FeB, Fe2B and finally Fe3C. The nitride and borides formed from the alloying element of Cr which is CrN $[20,24]$, CrB, and Cr₂B $[18]$, which agrees with the results of the observations under the optical microscope and SEM.

Fig. 5. XRD pattern of the treated sample at 520°C for 8h

This XRD pattern indicates that the borocarbonitrided phases were detected with variable values in diffracted intensity according to the duration of the treatment. When nitrogen increased, $α$ phase disappeared in the thicker layers of borocarbonitrided. Its contribution becomes less intense to the point of disappearing. While this result showed that the borocarbonitriding at 520°C allows the formation of a layer of high hardness on the steel. This is linked to the presence of Cr in this type of steel which facilitates the formation of high hardness phases. The latter exerts a significant action in terms of nitrogen and enrichment boron and hardening.

C. Microstructure observation

Fig. 6 shows the microhardness profile of the treated sample at 520°C for 8h. The Microhardness profile obtained from crosssections of the treated specimen, it shows the presence of a slope interface between the case (borocarbonitrided layer) and the core. This sample shows high surface microhardness values that drop decreasingly at the case/core interface to substrate microhardness values. On the other hand, Fig. 5 demonstrates higher near-surface hardness values between 500 and 850 HV, and large depth is obtained. Then, they continue to decrease in a very brutal way between 208 and 274 HV to reach the zone of the substrate. The hardness measured at the substrate level under an indentation load of 200g is practically constant, with an average value of 204 HV. This result is in good accordance with the previously observed in micrographic structures.

Fig. 6. Microhardness profile of the treated sample at 520°C for 8 h

The phenomenon of structural hardening observed during the borocarbonitriding of steel 30 CrMo12 is due to the affinity of nitrogen and boron for chromium, which causes the coherent precipitation of nitride of face-centred cubic structure and borides $(CrB \text{ and } Cr_2B)$ [18], which by opposing the dislocations movement cause significant material hardening. This result showed that the borocarbonitriding at 520°C allows to illustrate the formation of a high hardness layer on steel 30CrMo12. This is linked to the presence of chromium (Cr) in this steel. On the other side, the other elements are mainly used to fix the characteristics at heart at core. This layer is superior to that of the cemented, nitrided and borided layer simple.

IV. CONCLUSION

The microstructural and micromechanical characterizations of borocarbonitrided for low alloy steel 30CrMo12 were studied. The results obtained at 520°C for 8 h show that a hard layer of maximum thickness was formed to ensure a long life of these parts. The formed layer corresponds mainly to $ε$ -Fe₂₋₃N and γ'-Fe₄N of iron nitrides, nitroferrite (α), FeB, Fe₂B iron borides, as well as iron carbides Fe₃C. Also note the presence of chromium nitrides (CrN) and chromium borides (CrB) and $Cr₂B)$. It confirmed that nitrogen, enrichment boron and hardening dependent strongly on the following process parameters: time, temperature and the initial composition of steel. Among the alloying elements, chromium is the most important. It has been observed that increasing in nitrogen, boron, and carbon considerably increase the borocarbonitride layer and improve its mechanical properties.

REFERENCES

- [1] R. Lévêque, "Traitements et revêtements de surface des métaux", 2eme édition, Technique et ingénierie – EEA, 496 pages, Dunod, France, (2022), EAN13 : 9782100834020.
- [2] R. Gras, ʺ Traitements et revêtements de surface à usage tribologique ", Techniques de l'ingénieur Surfaces, vol. base documentaire : TIB463DUO., no ref. article : tri5100. Editions T.I., (2011). DOI: 10.51257/a-v1-tri5100
- [3] S. Sanjari, A. Habibolahzadeh, and S. Heydarzadeh, "Duplex Surface Treatment of AISI 1045 Steel via Plasma Nitriding of Titanized Layer." International Journal of Engineering, Transactions B: Applications, Vol. 28, No. 8, (2015), 1193-1198. DOI: 10.5829/idosi.ije.2015.28.08b.12.
- [4] O. Belahssen, A. Chala, S. Benramache, B. Djamelb, and Ch. Foued, "Effect of Gas Mixture H2-N2 on Microstructure and Microhardness of Steel 32CDV13 Nitrided by Plasma." International Journal of Engineering, Transactions A: Basics, Vol. 27, No. 4, (2014), 621-624. DOI: 10.5829/idosi.ije.2014.27.04a.13
- [5] H. Weil, S. Jégou, L. Barrallier, A. Courleux, and G. Beck, "Modélisation de la fatigue pour la nitruration gazeuse : méthodologie", Congrès Français de Mécanique, Lyon, 24-28, (2015). https://hal.archives-ouvertes.fr/hal-01176890
- [6] G. Fallot., "Rôle du carbone lors de la nitruration d'acier de construction et influence sur les propriétés mécaniques". Thèse ENSAM Aix-en-Provence, (2015). https://pastel.archives-ouvertes.fr/tel-01347211
- [7] L. Barrallier, "Thermochemical Surface Engineering of Steels, Chap.10 : Classical nitriding of heat treatable steel-Metals and Surface Engineering". Woodhead Publishing, 392-411, (2014).
- [8] A. Rezala, and M. Arbaoui, "Traitements de boro-Carbo-Nitruration en robotiqueʺ, Tribologie : approches scientifiques et applications industrielles, Actes des 24e Journées Internationales Francophones de Tribologie (JIFT 2012), Aix en Provence, France, (2012). https://www.pressesdesmines.com.
- [9] O. Belahssen, and A. Chala, "Microstructure of Low Alloyed Steel 32CDV13 Nitrided by Plasma", International Journal of Science and Engineering Investigations, Vol. 1, No. 11, (2012), 22-24.
- [10] S. Jegou, L. Barrallier, R. Kubler, and M. Somers, "Evolution of residual stress in the diffusion zone of a model Fe-Cr-C alloy during nitriding". HTM Journal of Heat Treatment and Materials : Vol. 66, No. 3, pp. 135 (2011). DOI:10.3139/105.110104.
- [11] F. Hakami, M.H. Sohi J.R. and Ghani, "Duplex surface treatment of AISI 1045 steel via plasma nitriding of chromized layer", Thin Solid Films, Vol. 519, No. 20, (2011), 6792-6796. DOI:10.1016/j.tsf.2011.04.054.
- [12] M. Heydarzadeh Sohi, M. Ebrahimi, A. Honarbakhsh Raouf and F. Mahboubi, "Effect of plasma nitrocarburizing temperature on the wear behavior of aisi 4140 steel", Surface and Coatings Technology, Vol. 205, Supplement 1, No. 0, (2010), S84-S89. DOI: https://doi.org/10.1016/j.surfcoat.2010.04.054.
- [13] A. Rezala, "Contribution à la compensation des erreurs statiques et dynamiques d'un robot manipulateur", Thèse de magister, 134p, univblida1, Algérie, (2008). http://di.univ-blida.dz:8080/jspui/handle/123456789/5180.
- [14] N. Krishnaraj, P.B. Srinivasan, K.J.L. Iyer and S. Sundaresan, "Optimization of compound layer thickness for wear resistance of nitrocarburized H11 steels", Wear, Vol. 215, No. 1-2, (1998), 123-130. https://doi.org/10.1016/S0043-1648(97)00276-7.
- [15] X. Tong, Z. Zheng, Z. Zhang and D. Liu, "The effect of epsilon phase on the friction and wear of nitrided die steels (Research note)." International Journal of Engineering, Vol. 11, No. 3, (1998), 175-179.
- [16] D. Ghiglione, C. Leroux and C. Tournier, " Nitruration, nitrocarburation et dérivés", Techniques de l'Ingénieur, M 1227, (1996). [https://www.techniques-ingenieur.fr/base-documentaire/archives](https://www.techniques-ingenieur.fr/base-documentaire/archives-th12/archives-traitement-des-metaux-tiamd/archive-1/nitruration-nitrocarburation-et-derives-m1227/)[th12/archives-traitement-des-metaux-tiamd/archive-1/nitruration](https://www.techniques-ingenieur.fr/base-documentaire/archives-th12/archives-traitement-des-metaux-tiamd/archive-1/nitruration-nitrocarburation-et-derives-m1227/)[nitrocarburation-et-derives-m1227/](https://www.techniques-ingenieur.fr/base-documentaire/archives-th12/archives-traitement-des-metaux-tiamd/archive-1/nitruration-nitrocarburation-et-derives-m1227/)
- [17] C. Leroux, "Cémentation par le carbone et carbonitruration Mise en œuvre des traitements", Techniques de l'ingénieur Traitements des métaux, vol. base documentaire : TIP553WEB., no ref. article : m1226. Editions T.I., (2011). DOI: 10.51257/a-v2-m1226.
- [18] Z. Nait Abdellah, "Caractérisation physico-chimique, calculs thermodynamiques des phases et simulation de la cinétique de croissance des couches de borures formées sur des substrats métalliques", Thèse de Doctorat, 120p, UMMTO, Algérie, (2012). https://www.ummto.dz.
- [19] M.A. Djouadi, C. Nouveau, O. Banakh, R. Sanjinés, F. Lévy and G. Nouet, "Stress Profiles and Thermal Stability of CrxNy Films Deposited by Magnetron Sputtering." Surface and Coatings Technology, Vol. 151–152, (2002), 510-514. https://doi.org/10.1016/S0257-8972(01)01635-8
- [20] C. Nouveau, M.A. Djouadi, P. Beer, P. Jacquet, L. Imhoff, R. Marchal and M. Lambertin, "Duplex Treatment Based on the Combination of Ion Nitriding and PVD Process: Application in Wood Machining", in International Wood Machining Seminar IWMS 15, Los Angeles CA, USA. (2001).
- [21] F. Borgioli, A. Fossati, E. Galvanetto and T. Bacci, "Glow-discharge nitriding of AISI 316L austenitic stainless steel: influence of treatment temperature", Surface and Coatings Technology, Vol. 200, No. 7, (2005), 2474-2480. https://doi.org/10.1016/j.surfcoat.2004.07.110.
- [22] A.M. Abd El-Rahman, F.M. El-Hossary, F. Prokert, N.Z. Negm, N. Schell, E. Richter and W. Möller, "In-situ stability study of nitrocarburized 304 stainless steel during heating", Surface and Coatings Technology, Vol. 200, No. 1-4, (2005), 602-607. https://doi.org/10.1016/j.surfcoat.2005.01.026.
- [23] M.E. Djeghlal, N. Hamedi and L. Barrallier, "Caractérisation métallurgiques et mécaniques de couches nitrurées, relation microstructure-comportement". J. Phys. IV France, Vol. 11, No PR10, (2001), Journées d'Étude des Équilibres entre Phases, Pr10-141-Pr10-145. https://doi.org/10.1051/jp4:20011021.
- [24] S. Jégou, "Influence des éléments d'alliage sur la genèse des contraintes résiduelles d'aciers nitrurés". Thèse, ENSAM Aix-en-Provence, Ecole Arts et Métiers ParisTech, (2009). https://pastel.archives-ouvertes.fr/pastel-00005632.
- [25] C. Ginter, "Influence des éléments d'alliage sur les mécanismes de nanoprécipitation et sur les mécanismes de durcissement d'alliages modèles (Fe-Cr et Fe-Cr-C) et d'aciers industriels nitrurés". Thèse Université de Nancy, (2006). https://www.researchgate.net.