Microstructural and micromechanical characterizations of the growing nitrided layer of 30CrMo12 steel

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Abstract— **The need to extend the service life of mechanical parts at a reduced cost has led to the use of thermochemical treatment to ensure surface properties with good core ductility of the treated parts. The objective of this study is to characterize the microstructure and micromechanics of the growing nitrided layer of 30CrMo12 steel frequently used in aeronautical construction. An experimental study on nitriding was conducted in which the steel used underwent a thermochemical nitriding treatment. The treated samples were characterized by optical microscopy, scanning electron microscopy, X-ray diffraction, and microhardness tests. The characterization results showed a considerable improvement in the surface properties (maximum hardness of 1119 HV and wear resistance) of the treated samples. The thermochemical nitriding treatment technique has the power to achieve a nitrided layer of maximum thickness, which increases the resistance to wear and friction as well as mechanical fatigue.**

Keywords—Nitriding, image analysis, microhardness, hardening, and 30CrMo12 steel.

I. Introduction

Presently, industrial competitiveness is a considerable asset, being able to produce products that do not wear out, are resistant to corrosion, and retain their mechanical, electrical, optical, or thermal properties…

The need to increase the durability of parts and reduce material costs have made the development of thermochemical treatments [1-5] an often essential option in the manufacture and preventive and curative maintenance of mechanical parts.

One of these thermochemical treatments is nitriding [6-8] which enriches the surface with nitrogen for surface hardening. This process is used to ensure a long life of mechanical parts.

The main of present study is to characterize the microstructure and micromechanics of the growing nitrided layer of 30CrMo12 steel, frequently used in aeronautical construction, using the experiment conducted using image analysis techniques (optical microscopy, scanning electron microscopy, X-ray diffraction) and microhardness profiles. The characterization results obtained showed a considerable improvement in the surface properties of the treated samples. This process can be implemented in a reproducible industry while allowing automatic control of the installations.

II. Experimental study

A. Presentation of materials

Low alloy steel 30CrMo12 [9] is chosen for nitriding treatment. This grade is selected from the most commonly used grades industrially in the manufacture of mechanical parts subjected to high fatigue stress, such as transmission gears on helicopter rotors. Its chemical composition was analyzed by Energy Dispersive Spectroscopy (EDAX) as shown in Table 1.

TABLE I. CHEMICAL COMPOSITION OF THE STEEL USED (WT .%)

Material	**C**	**Si**	**V**	**Cr**	**Mn**	**Fe**	**Mo**
30CrMo12	0.30	0.31	-	3.02	0.49	93.13	0.43

B. Processing procedure

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.

C. Nitriding treatment

The process used is gas nitriding [10]. The principle is as follows: the samples are placed in a controlled atmosphere oven where they are heated to a temperature of 520°C and into which gaseous ammonia is introduced which, under the effect of the temperature, decomposes according to reaction (1) to give atomic nitrogen N and gaseous hydrogen H2.

$$NH_2\,(g) \implies \mathrm{N}\,(at) + 3/2\ \mathrm{H}_2\,(g) \qquad (1)$$

Atomic nitrogen (N) will be absorbed by the alloy to form nitrides in its surface layers.

The experimental conditions adopted for gas nitriding are shown in the table below.

TABLE II. NITRIDING PARAMETERS FOR THE STEEL USED

Steel	***T_N (°C)***	***t_N (h)***	***Dissociation rate τ_N (%)***
30CrMo12	520	70	15

D. Characterizations

Optical microscopy

After nitriding treatment, the samples are cut, coated to avoid edge effects, and mechanically polished to diamond paste (0.05 µm). They are then observed using a NIKON Eclipse LV100 optical microscope equipped with a camera and connected to a microcomputer after an attack with Nital of composition (3% nitric acid, 97% ethyl alcohol). This chemical attack is carried out at room temperature, to highlight the structure of the matrix and the nitrided depth.

Scanning electron microscope

The JOEL 5600LV scanning electron microscope is used to obtain images modulated by secondary electrons (topographic contrast) or by backscattered electrons (atomic number contrast). The sample-detector working distance chosen is 8 mm and the acceleration voltage ranges from 5 kV to 20 kV. The probe current has been reduced to a minimum (10-10-10-11 A) to have a higher resolution. Finally, coupled with an X-ray spectrometer, the SEM allows chemical analyses to be carried out on small volumes (about 1 µm3) or to obtain images that give the special distribution of the elements (X-ray image). The samples were prepared in the same way as for metallographic observations.

X-ray diffraction analysis

DRX characterization of the crystalline phases was performed on a BRUKER D8 Advance diffractometer equipped with a linear localization detector. The diffraction angle 2θ is used to define the Bravais lattice of the phases and defines the crystallographic planes responsible for the beam deflection. The samples were perfectly polished to depths ranging from 0 to 300 µm, in steps of 10 µm.

Micromechanical analysis

The micromechanical analysis is performed using the classical method of microhardness measurement. Microhardness measurements are performed using an OPL Vickers microdurometer under a load of 200 g (Vickers scale HV0.2). The distance between two indentations is on average about thirty micrometers. They are measured on the cross-sections of the nitrided samples. The sample preparation is the same as for micrographic observations.

III. Results and Discussion

A. Metallographic observations

Optical microscopy performed on a cross-section of the 30CrMo12 sample nitrided at 520°C shows the morphology of the nitrided layer (Fig. 1). Successively less rich phases are encountered, as predicted by the Fe-N equilibrium diagram. The nitrided layer comprises the ε phase on the surface followed by γ` which constitute the combination layer (white in color), then the α phase (nitro ferrite) diffusion layer where the nitrides of the addition elements are found, then the base metal. The whitish appearance is due to the Nital attack of 3% nitric acid and 97% ethyl alcohol.

The structure of the nitrided zone therefore corresponds to:

- The thin white outer zone, not attacked by Nital, called combination layer.
- The intermediate zone is blackened by the Nital attack, which corresponds to the diffusion layer.
- Area of the core that corresponds to the base metal.

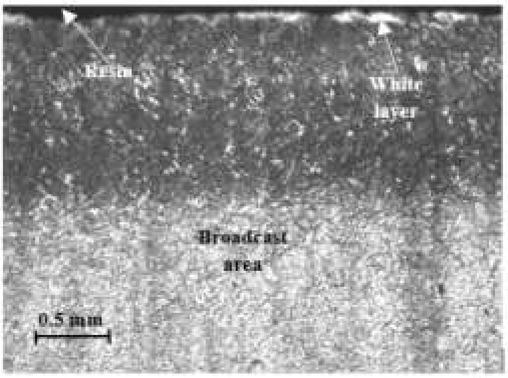

Fig. 1. Optical micrograph of a nitrided layer of 30CrMo12 steel.

Fig. 2, obtained by SEM from a cross-section of the 30CrMo12 sample nitrided at 520°C, confirms the results obtained by optical microscope.

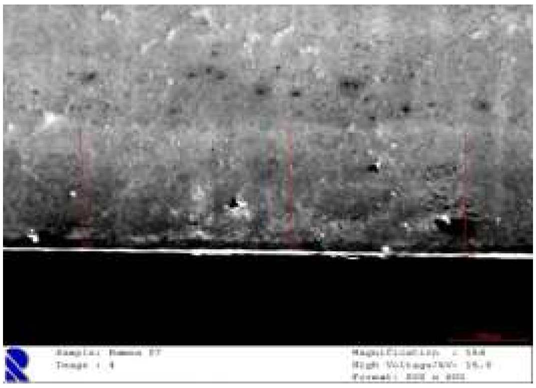

Fig. 2. SEM micrograph of the nitrided layer of 30CrMo12 steel.

B. Phase analysis using X-ray Diffraction

Fig. 3 shows the diffraction spectra of the phases of the 30CrMo12 steel nitrided at 520°C. The diffraction spectra allow to identify the phases present in the nitrided layer predictable from the equilibrium diagram. These spectra show the existence of the iron nitride ε ($Fe_{2\text{-}3}N$) diffracting along the planes (-1-11) and (-1-12); of the nitrides γ` along (100), (110), (111), (200), (210); the phase α along (200) and finally the nitride formed from the alloy element Cr which is CrN (111), (200) and (220).

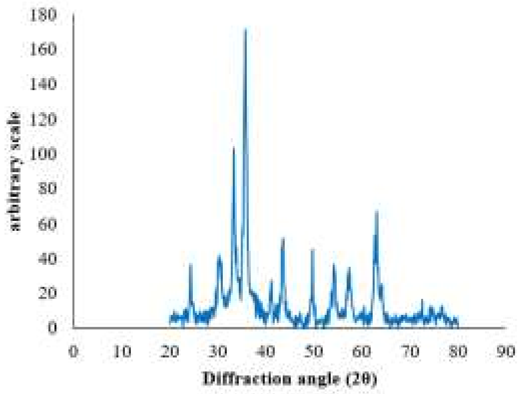

Fig. 3. Diffraction profile of 30CrMo12 nitrided steel.

C. *Micromechanical characterization of the nitrided layer*

The nitriding microhardness profile has an appearance in which three zones can be distinguished (Fig. 4):

- Superficial zone I corresponds to a maximum hardness of 1119 HV.
- Zone II corresponds to a level where the hardness decreases sharply, between 623 HV and 296 HV.
- Zone III is the core of the material not affected by nitriding; its hardness is constant 234 HV.

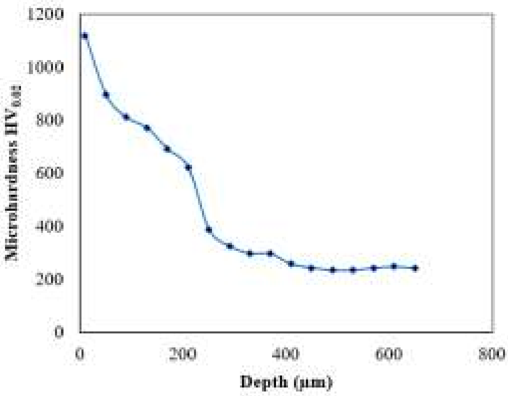

Fig. 4. Microhardness profile of 30CrMo12 nitrided steel.

The results of microhardness measurements of samples that have undergone nitriding treatment show an increase in surface hardness. The structural hardening phenomenon observed during nitriding of 30CrMo12 steel is due to the affinity of nitrogen for chromium which causes the coherent precipitation of face-centered cubic nitride and opposes the movement of dislocations, resulting in significant hardening of the material.

IV. Conclusion

The various results obtained during this study are summarized as follows:

- Superficial X-ray diffraction demonstrated the formation of the iron nitride ε ($Fe_{2\text{-}3}N$) phase, γ`-nitride, α-phase, and CrN-nitride.
- The γ` nitride shows a preferred orientation relative to the matrix, an observation confirmed by the SEM.
- The microhardness profile showed an increase in surface hardness of 1119 HV.
- Structural hardening depends on the presence of chromium in the alloy.
- The resulting nitrided layer improves the tribological properties.
- The appearance of a nitrided phase depends on the choice of the chemical composition of the substrate to be treated.

References

[1] A. Rezala, "Tribological interests of borocarbonitruration in robotics", The 5th International Seminar on Avanced Mecahnical Technologies SITMA'24, Tlemcen-Algeria, April 2024. [Online]. Available: https://sitma2024.univ-tlemcen.dz/sitma2024/

[2] A. Rezala, "Contribution to improving the mechanical characteristics of materials used in the structural components of industrial robots by borocarbonitriding treatment", 2nd National Conference on Materials, Energy & Environemental (NCMEE'2024), Biskra-Algeria, February 2024. https://doi.org/10.13140/RG.2.2.22540.28805

[3] A. Rezala, "Étude expérimentale des comportements mécaniques des surfaces de contact des robots industriels", 2nd International Seminar on Industrial Engineering and Applied Mathematics (ISIEM'22), Skikda-Algeria, October 2022. [Online]. Available: https://www.calameo.com/books/007244303c6879e030b41

[4] R. Gras, "Traitements et revêtements de surface à usage tribologique", Techniques de l'Ingénieur, TRI5 100 v1, May 2019. [Online]. Available: https://doi.org/10.51257/a-v1-tri5100

[5] R. Lévêque, and M. Colombié, "Traitements et revêtements de surface des métaux", 2e édition, Technique et ingénierie, Dunod, France, January 2022, 496 pages. EAN : 9782100834020, [Online]. Available: https://www.dunod.com/sciences-techniques/traitements-et-revetements-surface-metaux-1

[6] C. Leroux, "Nitruration et nitrocarburation - Matériaux mis en œuvre et propriétés d'emploi", Techniques de l'Ingénieur, M1228 v1, June 2012. [Online]. Available: https://doi.org/10.51257/a-v1-m1228

[7] N-E. Benrabia, "Etude thermodynamique de l'équilibre des phases dans les zones nitrurées, cas de la nitruration gazeuse des binaires et des aciers", Thèse de doctorat, Métallurgie, Ecole nationale polytechnique, Alger, 2018. [Online]. Available: http://repository.enp.edu.dz/xmlui/handle/123456789/1320

[8] O. Skiba, "Développement d'un procédé de nitruration pour l'aéronautique. Étude des mécanismes de durcissement sur des alliages fer-chrome nitrurés", Matériaux, Université de Lorraine, France, 2018. NNT: 2018LORR0099. [Online]. Available: https://hal.univ-lorraine.fr/tel-02006456

[9] A. Rezala, "Improvement of Surface Properties of 30CrMo12 Steel Using Borocarbonitriding Treatment", the 1st International conference on Materials Science and Applications ICMSA'23 (Hybrid), Khenchela-Algeria, February 2023. [Online]. Available: https://icmsa2023.sciencesconf.org

[10] Y. Zhang, "Nitruration gazeuse de l'acier à outil M2 pour amélioration des propriétés mécaniques de surface des molettes de roulage", Thèse de doctorat, Mécanique – matériaux, Paris, HESAM, December 2022. [Online]. Available: https://theses.fr/2022HESAE064