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The influence of carbon potential after gas-carbonitriding on the microstructure and fatigue behavior of low alloyed steel

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Abstract

In this paper, the effects of gas-carbonitriding time on fatigue limit improvement of low alloy steel specimens were investigated by experimental tests of three points fatigue flexion. Besides, metallurgical evaluations and micro-Vickers hardness tests were performed employing metallographic techniques, optical, scanning electron microscopy and x-ray diffraction techniques. Test findings showed that this amount remarkably improved the components fatigue resistance. The fatigue life of carbonitrided specimens was prolonged between 16%, for CN11 (i.e. austenization-870°C/7 h, atmosphere with 1% carbon potential, quenching-oil medium and tempering-200°C/1 h), and 32%, for CN21 (i.e. austenization-870°C/8 h, atmosphere with 1, 2% carbon potential, quenching-oil medium and tempering-200°C/1 h), compared to the untreated state. It is obvious, from the fractography analysis, utilizing (SEM), that fatigue cracks appeared first at the carbonitrided specimens surface.

Introduction

Machine components operating under cyclic loads can be influenced by fatigue failure. The improvement of fatigue strength for those made up of low-alloyed steels results from the hardening treatments producing a hardened layer on a ductile core. Within this framework, the application of thermo chemical surface treatment enhances the strength of steels and the hardness of the surface [1–3]. Generally, case hardiness may be considerably increased through the diffusion of carbon and/or nitrogen into the surface, applying thermo chemical treatments (carburizing [2, 4, 5], carbonitriding [6, 7], nitrocarburizing [8] and nitriding processes [9–12]. In particular, carbonitriding has been employed in order to improve fatigue and wear resistance of steels utilized in many parts of machines [13], such as cam shafts, screws, engine valves and gears crank shafts as well as plates of various kinds of machines, for the purpose of enhancing fatigue strength and increasing wear resistance [14]. The improvement of the steels fatigue life through carbonitriding strongly depends on the carbonitrided layers characteristics, especially the work-hardening retained austenite and residual stresses distributions [15]. Carbonitrided steel is also related to retained austenite and martensite microstructures in proportions depending on the conditions of carbonitriding and the kind of steel. Such variation of microstructure enhances the treated components fatigue strength with the rise of the number of cycles to failure or the endurance limit [16–18].

Carbonitriding is a surface hardening process. It is characterized by the diffusion of carbon and nitrogen into the component’s surface in the temperature range 850°C–880°C. The first step is saturating the surface in carbon and nitrogen, then the hardening and tempering steps. The hard carbonitrides, nitrides and carbides layer formed at the surface ameliorates the fatigue strength [19, 20]. The carbonitriding treatment results in the formation of hardness gradient and the distribution of the residual stresses with compressive stresses at the surface microstructure because of the variation of volume during the martensitic transformation [21, 22]. The surface layer increased hardness and strength, together with the compressive stress caused by the interaction between the surface and the core, improve the surface properties and raise the resistance to fatigue [23, 24].
AISI 4340 steel is employed to make gears in automobile industry and carbonitriding is often used to increase hardness and modify fatigue. The purpose of this work is to study the effect of carbon and nitrogen potentials on the fatigue behavior of gas-carbonitrided under a three-point bending and identifies fatigue mechanisms.

The influence of surface modifications resulting from gas-carbonitriding is also examined employing a micro-hardness tester, optical microscopy, scanning electron microscopy (SEM) with x-ray diffraction (XRD) and three-point bending fatigue machine. The failure mechanisms are finally analyzed and discussed.

Material and experimental procedure

The investigated material, having the chemical composition of (wt%) 0.26 C, 0.84Mn, 1.06Cr, 0.3Si, 0.22 Mo, 0.0097 P, 0.029 S and balance Fe, is an AISI 4130 steel manufactured in bars from similar cast. The diameter of these bars is equal to 20 mm. It is generally utilized in the field of mechanical industry to manufacture automotive transmission parts.

Two fractions of retained austenite were obtained in the hardened layers through the application of two gas-carbonitriding times to notched fatigue specimens (Kt = 1.6) (figure 1). The chosen notch represents gears teeth geometry. The specimens were carbonitrided at 870 °C for 7 and 8 h in an atmosphere of endothermic gas containing a high amount of methanol gas and ammonia gas mixture to diffuse nitrogen and carbon simultaneously into the tested steel. Subsequently, they were quenched in oil at a temperature of 60 °C and tempered at 200 °C for 60 min. Table 1 shows the treatment parameters.

After being polished at room temperature, the untreated and treated specimens’ cross-sections were etched in 4% Nital solution. Subsequently, they were observed using an optical microscope. The measurement of compressive residual stress and retained austenite profiles obtained by the two treatments were specified employing x-ray diffraction of the martensite and the retained austenite using a Pulstec μ-X360 apparatus. The retained austenite volume fraction was measured using x-ray diffraction. The depth profiles were obtained with the removal of successive layers employing electrochemical polishing. More information about the measurement of retained austenite and residual stress is listed in table 2. The evaluation of surface hardening resulting from the different carbonitriding treatments was carried out utilizing micro hardnesstests under a load equal to 50 gf.

Three-point bending fatigue tests were performed on notched specimens (Kt = 1.6) to assess the effect of the structure of the carbonitrided layers and their crack resistance on the endurance limit. Limited to a number of cycles of 10⁶, they were execute data frequency equal to 15 Hertz with stress ratio R1 = 0.1. Then SEM was used to observe the fatigue fractured surfaces and analyze crack initiation position.

Table 1. Gaseous carbonitriding parameters and specimen designations.

| Specimen designations | Gas components | Carbon potential (%) | Temperature (°C) | Time (h) | Tempering parameters |
|-----------------------|----------------|----------------------|------------------|----------|----------------------|
|                       | CH₄ + NH₃      | 1                    | 870              | 7        | 200                  |
|                       | CH₄ + NH₃      | 1.2                  | 870              | 8        | 200                  |

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Structure

Figure 2 shows typical transverse surface microstructures of the carbonitrided samples are presented. After the metallographic procedure of the samples, which consists in polishing and chemical etching (Nital 4%), the obtained microstructures were observed using an optical microscope (figure 2). In the two thermochemical treatments, typical transverse surface microstructures of the carbonitrided samples were observed, which reflects the presence of a martensitic microstructure. Retained austenite was in white colour. Obviously, this soft phase minimized the surface hardness remarkably and changed the residual stresses distribution. Adding ammonia gas to the atmosphere in the carbonitriding procedure reduced the internal oxidation. Another quite dense black under layer, enriched with nitrogen and carbon, was also revealed. This layer is characterized with martensitic needles including huge quantity of retained austenite (untransformed) and Fe₃C carbides. The samples x-ray diffraction patterns are presented in figure 3. These samples were treated according to two carbonitriding conditions. It is noticed that γ'(Fe₄N) and ε(Fe₂₋₃CN) precipitates occurred in the supersaturated solid solution of nitrogen and carbon (martensite and retained austenite).

Retained austenite and residual stress evaluation

The depth profiles were obtained with the removal of successive layers applying electrochemical polishing. The obtained results showed that dispersion was not high. As far as the same depth of treated layers is concerned, the retained austenite fractions, obtained after CN11 treatment, were more important than those obtained after CN21. The following conclusions can be drawn from the measurement of the retained austenite profiles of the specimens (figure 4):

- At the hardened layer, the amount of retained austenite provided by the CN11 treatment exceeded that provided with the CN21 treatment.
- The maximum retained austenite fractions of 16% and 18%, respectively, for CN11 and CN21 treatments were attained at a depth equal to 50 μm.
- Close to the surface, a progressive decrease of the retained austenite fraction to 2% and 3% was observed, respectively, for CN21 and CN11 treatments.

The results of the x-ray diffraction measurements of the residual stresses in the martensitic phase of the carbonitrided layers revealed almost super-imposed profiles on the appearance of the retained austenite profiles. For both treatments, the measured residual stresses profiles showed a compression state in the carbonitrided layers (figure 5). Surpassing this depth, the highest compressive residual stress was about −160 MPa for CN11 treatment, and −310 MPa for CN21, treatment. These values were attained respectively at depths of 300 μm and 250 μm for the CN11, and CN21 treatments. The depth of transformation from compressive to tensile residual stresses was almost equal to the case depth obtained in the hardness profile.

Hardness profiles

Figure 6 compares the micro-hardness profiles in the AISI 4130 steel surface layer. This steel was carbonitrided at 870 °C for 7 h (CN11) and 8 h (CN21). The distance beneath the surface is considered as the samples effective

| Diffraecometer : Pulstec μ-X360 apparatus |
| Retained austenite determination |
| Austenite γ face centered cubic {200} plane, 2θ = 78.99° |
| {220} plane, 2θ = 128.70° |
| Mesure uncertainly ±0.3% |

| Residual stress determination |
| Detector 2D |
| Lattice plane {211} |
| Radiation Cr (λ = 2.291 Å) |
| Bragg angle 2θ = 156.4° |
| Exposure time 30 s |
| Mesure uncertainly ±10 MPa |
case depth while hardness is at least 550 HV0.05. Another factor that is taken into account when measuring case depth is the martensitic structure. Steady micro-hardness values evolution with the observed micro-structure was noticed as a function of layer depth. As a result, the micro-hardness profile for CN21 specimen showed a

Figure 2. Cross-sectional upper surface microstructures after carbonitriding (a) CN11 and (b) CN21.

Figure 3. X Ray diffraction specters for carbonitrided specimens for : CN11 and CN21.
value of 865HV0.05 at the surface as a maximum and 535HV0.05 at the core. However, for the CN11 the obtained values were 770HV0.05 and 530HV0.05 for the surface and core, respectively. Such discrepancy in the values of micro-hardness for both specimens may be due to the decomposition of tempered martensite and stress relaxation [25, 26].

Fatigue behavior of carbonitrided steel

Figure 7 represents $S-N$ curves of both untreated and carbonitrided AISI4130. Table 3 demonstrates the endurance limits for $10^6$ cycles obtained by the staircase technique and the improvement caused by both kinds of treatment. The carbonitriding treatment significantly ameliorated the fatigue response, revealing the enhancement range provided by previous tests carried out on steels. The endurance limits of the untreated specimens and the carbonitrided specimens 640 MPa, 740 MPa (for CN11) and 840 MPa (for CN21) correspond respectively to 16% and 32% enhancement, compared to the untreated state. In fact, according to the literature, fatigue strength improves with the increase of hardness and retained austenite [6, 7].

Fatigue fracture

Figure 8 depicts the fatigue fracture mechanisms of the carbonitrided samples and untreated state. As far as the latter is concerned, the appearance of fatigue cracks at the surface of the specimens was irrespective of the applied stress level (figure 8(a)). Stress concentrations, for instance, took place at grooves or scratches developed by surface machining. Indeed, microfractographic analysis of the fracture surfaces of carbonitrided samples, under low magnification, showed different mechanisms of fracture. (figures 8(c) and (e)) reveals fatigue fracture
Figure 5. Residual stresses with different conditions: (a) profiles as a functions of the depth. (b) Surface and maximum value.

Figure 6. Microhardness depth profiles with different conditions.
Figure 7. Fatigue life improvement before and after gas-carbonitriding: wohler diagrams (b) fatigue limit for different conditions.

Table 3. Summary data for study different conditions.

| State    | HV 0.05 | Case depth (μm) | γτ (%) | στ (MPa) | ΔσD (MPa) | Improvement (%) |
|----------|---------|-----------------|--------|----------|-----------|-----------------|
| untreated | 270     | —               | —      | —        | 640       | —               |
| CN11     | 770     | 1020            | 13.5/18.4 | −67/−165 | 740       | 16              |
| CN21     | 865     | 1140            | 9.4/15.8 | −160/−310 | 840       | 32              |
mechanism. The crack initiation sites in the carbonitrided specimens were at the surface in the two domains (HCF, LCF).

These nucleations were absolutely observable at the sample surface in the notched zone close to the rupture, in which micro-fractures, perpendicular to the highest tensile stress produced by the ending fatigue, appeared. Thus, the control of the life of carbonitrided parts was essentially done by the compound layers resistance to the initiation of fatigue cracks. Analysis of the fracture surface at the bottom of the notch applying SEM shows that micro-cracks appeared on the surface in several planes due to the mechanism of crack-initiation. The coalescence of these microcracks produced a macroscopic crack, which caused the appearance of the main fracture. The microfractographic analysis demonstrates that higher magnification favored intergranular cracks and initiated intergranular carbide precipitation and internal oxidation (figure 9). In fact, the grains facets were sprinkled with different-sized particles (figure 10). The fracture surface overall aspect reveals continuous brittle fatigue crack propagation in the treated layer. The brittle characterizing this fracture resulted from the

Figure 8. Micrographs of the fracture surface for: (a) fatigue fracture surface aspect of untreated state ($\sigma_{\text{max}} = 421$ MPa; $N_r = 177437$ cycles), (b) detail view untreated state, (c) fatigue fracture surface aspect of carbonitrided AISI 4130 steel CN11 ($\sigma_{\text{max}} = 984$ MPa; $N_r = 8100$ cycles), (d) detail view treated state CN11, (e) fatigue fracture surface aspect of carbonitrided AISI 4130 steel CN21 ($\sigma_{\text{max}} = 865$ MPa; $N_r = 25070$ cycles), (f) detail view treated state CN21.
intergranular rupture on the surface where fatigue crack progressively shifted to transgranular fracture in the under layers (figure 11). Fatigue striations appeared just at the interface separating the treated layer and the bulk material (figure 12).
Discussion

Based on the obtained results, during treatment, various complex transformations happened in this steel. Indeed, many factors affected the mechanical properties of gas-carbonitrided steel. Among these factors, the presence of retained austenite, hardness, surface finish, chemical composition, microstructure as well as residual stress were investigated. This study demonstrates the effect of the carbonitriding treatment times on the superficial hardening gradient resulting from the heterogeneous microstructure gradient (retained austenite and fine martensite). The austenite-martensite transformation, during cooling, generated new dislocation blocking interfaces and therefore stacking interfaces and dipoles which hardened the retained austenite. Specific carbides and retained austenite appeared in the structure during the heat treatment of steels with increased amount of carbon and more alloy elements, which shifted Ms and specifically Mf temperatures to reduced values. These carbides as well as their morphologies rely on the composition of the alloy and the processing time utilized to form the final structures. Obviously, the presence of Fe3C carbides, rich in alloy in γ elements (molybdenum and chromium), improved the fatigue resistance and the static strength of steel components [18]. Indeed, the carbon
potential on the surface was more than 0.8% and the rise in the holding time minimized it towards the sub layers. Therefore, as previously mentioned, the reduction in carbon content resulted in the increase of Ms and Mf, leading to a substantial rise of martensite content and to the decrease of retained austenite content when the samples were under quenching treatment. Besides, especially at high temperature, the reduction in carbon potential caused the destabilization of austenite during quenching which favored the carbides precipitation in accordance to the chemical composition of as-studied steel. This study proved the importance of the retained austenite content and its state of hardening in the carbonitrided layers. Therefore, any evolution of the retained

Figure 12. Fatigue striations: (a) untreated state, (b) CN11 state and CN21 state.
austenite can modify the hardening of the treated layer. Since the microstructure of the martensitic phase strongly depends on its carbon content in solid solution, the knowledge or the approximation of the carbon and nitrogen profile imposed by the carbonitriding is essential to understand the resulting hardening. The high microhardness value of 865 HV0.05 of the CN21 sample, compared to that of CN11, may be attributed to the retained austenite rate. The same core hardness for the studied samples, put randomly in the range of (320–535) HV0.05, suggests that no effect of the carbonitrided time was noticed on the substrate hardness. Carbonitriding and subsequent quenching-tempering increased the fatigue resistance and the retained austenite content effect was particularly important. Within this framework, the efficiency of the retained austenite phase remains the most crucial issue to deal with [16]. The saturation of surface layer is usually attained with carbon to a eutectoid or quite hypereutectoid content. The microstructure, after quenching, comprised high-carbon martensite and ultimately small quantity of carbides. Carbides were obtained at increased temperatures. In fact, high carbon content led to the appearance of the carbonitrided layer. In this study, the sufficient quantity of molybdenum and chromium associated to the involved nitrogen resulted in the formation of the corresponding carbonitrides.

The enhancement of fatigue strength of the carbonitrided AISI 4130 steel was 32%, for CN21 treatment, and 16% for CN11 treatment, in comparison to the untreated steel fatigue strength. This discrepancy may be due to the role of the distribution of residual stress around the crack initiation region and the microstructure. In fact, the micro-fractographic analysis of the fatigue rupture surface reveals that crack propagation accelerated after initiation. Based on Wohler diagram flattened shape, we assume that crack initiation phase is the major factor of the fatigue life [27, 28]. This crack initiation can be attributed, in major part, to the microstructural characteristics, as summarized in table 3. In surface layers, the compressive residual stress distribution and microstructure favored the CN21 treatment more than the CN11 treatment. These findings show the enhancement of the fatigue strength. This improvement associated to the retained austenite fraction developed during cyclic loading as well as its influence on the deformation.

Conclusions

This research work has shown that the two gas carbonitriding treatments play an important role in improving the mechanical properties of AISI 4130 steel. These treatments led to the variation of micro-hardness and residual stress. These changes resulted essentially from the retained austenite fraction in the microstructure. It was also proven that during carbonitriding, good microstructure containing less retained austenite together with martensite may be formed in the surface layer. The change of microstructure increased the micro-hardness of the carbonitriding layer. The martensitic layers concurrent enrichment in carbon and nitrogen affected the hardenability and toughness trends of the AISI 4130 steel. Accordingly, the development of three different layers enhanced the fatigue behavior. Fatigue strength improvements of the carbonitrided steel, in comparison to its untreated state, were about 32%, for the CN21 treatment, and 16% for CN11 state treatment. They showed the highest retained austenite fractions equal to 16% and 18.5%, respectively. This enhancement is due to the superficial work-hardening positive effects and the important level of austenite retained at the notch.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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