Fatigue Behavior and Fracture Mechanisms of Nitrided Nodular Cast Iron

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ABSTRACT. *The fatigue behavior of nitrided nodular cast iron is examined in this contribution. Special attention is given to the quantification of the fatigue improvement and the investigation of the link between microstructure and fracture mechanisms. Rotary bending fatigue testing shows a significant increase in fatigue limit and a dual trend in the S/N curve. Therefore, selected fracture surfaces and fatigue crack paths were investigated to determine fracture mechanisms associated to the different fatigue lives. Factors such as structure of nodular cast iron, content of carbides, penetration depth and concentration of nitrogen on the boundaries of ferritic grains below the white layer were found relevant. An interpretative model of the fatigue response of nitrided nodular cast iron is proposed.*

INTRODUCTION

Gas nitriding is a thermo chemical treatment commonly used to enhance wear, fatigue and corrosion properties of mechanical components, such as gears, crankshafts, dies and tools, with minimal distortion [1]. Gas nitriding involves the adsorption of atomic nitrogen into the material surface and its subsequent diffusion inside the part. The nitrogen-rich surface layer (i.e. case) is relatively thin (i.e. < 500 μm), it is harder than the core material and it is subjected to compressive residual stresses. The properties of a nitrided component are determined by both core strength and case characteristics (i.e. structure of compound layer and the diffusion zone).

Prior investigations on nitrided steels have revealed that the increase in fatigue limit is very significant with a dependence on case depth of nitrided layers and a strong notch effect, [1-3]. Long lives of nitrided specimens were often associated to fatigue crack initiation from subsurface defects rather than the classical free surface. Models predicting the fatigue strength of nitrided steels have been proposed by regarding the strength and residual stress distribution in the cross-section of specimens [4].

The application of nitriding to nodular cast iron (NCI) is, differently from steels, not extensively documented. NCI is a widely used construction material in the fabrication of severely stressed mechanical parts of complex geometry because it combines the cost-effective casting technology with high fatigue strength, [5]. The microstructure of nodular cast iron is characterized by a distribution of spherical nodules of graphite in a metallic (i.e. pearlitic or/and ferritic) matrix. The nitriding process applied to NCI shares characteristics of the carbonitriding process of construction steels because of the high carbon content of NCI favors the formation of carbonitrides in the case, [6, 7]

This paper presents the fatigue response, the hardened layer structure and the fatigue fracture mechanisms of a ferritic NCI (i.e. EN - GJS 400) upon application of a patented gas nitriding treatment (Nitreg®). The objectives are: i) the quantification of the fatigue improvement obtained by nitriding ii) a discussion of the observed scatter in the nitrided test data iii) advancement of ideas for a fatigue life prediction model.

MATERIAL AND EXPERIMENTAL PROCEDURES

The test material was a ferritic nodular cast iron EN - GJS 400 with chemical composition according to the EN 1564 norm. The mechanical properties of the untreated ferritic NCI were: R_m = 450 MPa, A = 19 %, E = 168 GPa. Two sets of smooth fatigue specimens were prepared by machining from castings. One set was used to obtain the reference fatigue response of NCI; the other set of specimens was subjected to a nitriding treatment by the patented Nitreg® Controlled Potential process (Nitrex, USA) prior to fatigue testing. The structural analysis was performed on polished and etched specimen cross sections taken from broken specimens in the optical light metallographic microscope according to the norm and by the methods of quantitative metallography,[8]. The base microstructure is characterized by a ferritic matrix (average ferrite grain size d_m = 44 μm) with a regular distribution of graphite spherical nodules with size ranging from 30 to 60 μm and a nodule count of N = 197 nodules/mm^2. A discontinuous network of carbides with different quantity of carbides on the boundaries of eutectic cells in the ferrite matrix was also observed. The nitrided layer was analyzed using color etching techniques [9] and carbides and the nitrogen distribution in the case were characterized by EDS analysis [10]. Fig.1 shows a thin white (compound) layer on the surface, a diffusion zone and a sub - diffusion zone. The white layer was continuous with variable thickness from 10 to 28 μm and with local presence of graphite particles.

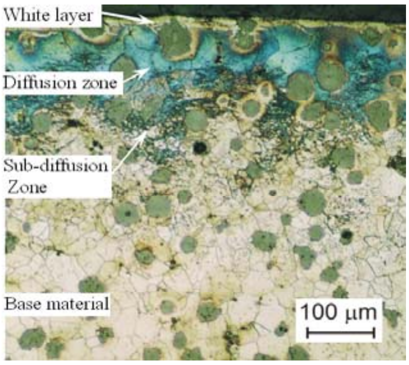

Fig. 1 – Microstructure of the nitrided layer, etched with 5 % molybdenum acid

The hardness profile characterizes the effectiveness of the nitriding treatment and is used to define an effective nitrided depth. Microhardness (HV 0.2) at different distances from the surface was measured on metallographic cross sections taken from selected fatigue specimens (i.e. 3 and 4 indicated in Fig. 2) to investigate specimen-to-specimen local changes in hardness pattern. Microhardness decreases from about 600 HV 0.2 with increasing distance from surface to 178 HV 0.2 corresponding to the hardness of the ferritic NCI. The effective case depth (defined as the position where the local hardness is 10 % above the core hardness) was found to be 0.4 mm for both specimens. However, specimen 3 has a softer case than specimen 4. Reference data were obtained by a second method of hardness profile characterization consisting of surface microhardness measurements followed by sequential layer removal. Fig. 2 shows that the trend of both hardness profile measurement techniques is similar. However, the layer removal method yields a slightly smaller case depth (i.e. 350 μm) and a steeper gradient than the cross sectional measurements.

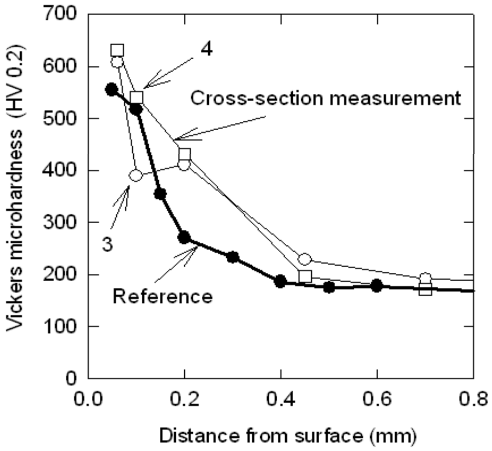

Fig. 2 – Microhardness profiles in the surface layer

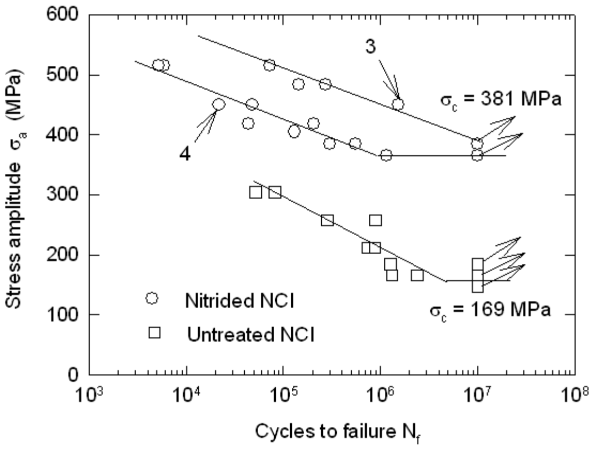

Fig.3 - Fatigue response of NCI in the untreated and nitrided conditions

The fatigue S/N curves for the untreated and the nitrided NCI were obtained using smooth 6-mm-dia specimens on a rotating-bending testing machine operating at 50 Hz (i.e. load ratio R = -1). A standard procedure for fatigue curve and fatigue limit determination at 50 % probability of survival using a limited number of specimens was adopted [3]. Tests were interrupted at 10^7 cycles if the specimen did not fail. The fatigue limit σ_c was determined according to a reduced staircase method [3]. The fatigue fracture surfaces of selected specimens were investigated in the SEM to identify fatigue initiation location and the mechanisms of stable crack propagation.

RESULTS AND DISCUSSION

Fatigue response of nitrided nodular cast iron

The S/N curves of the untreated and nitrided NCI are shown in Fig.3. The fatigue limit is σ_c = 169 MPa in the untreated NCI and σ_c = 381 MPa for the nitrided NCI. The improvement in fatigue limit of NCI upon nitriding is therefore higher than 100 %. This increase is in line with the results determined by previous tests on steels [3]. Inspection of Fig. 3, however, shows also that the scatter in fatigue life of the nitrided NCI data can be rationalized using two distinct parallel trend lines. The two trend lines connect data associated possibly to different fracture mechanisms. For example, specimens denoted as number 3 and 4 were subjected to the same applied stress amplitude but their fatigue lives differed by more than two orders of magnitude. This observation reminds that in steels a similar dual trend in fatigue data of nitrided specimens is associated with fatigue crack initiation at internal hard inclusions and that the subsurface initiation is typically associated to long lives and relative low stress amplitude, [3, 4]. Therefore an explanation of the nitrided data of Fig. 3 is sought inspecting fatigue fracture surfaces for the evidence of subsurface initiation.

Fatigue fracture surfaces

The macrofractographic view of nitrided specimens tested at the same stress level and having very different fatigue lives (i.e. specimens 3 and 4) revealed similarities in the fatigue fracture process: both surfaces are formed by two concentric areas, the light annular area of the stable crack propagation and the central dark area of the final fracture. In nitrided steels, the activation of the subsurface initiation mechanism is frequent and associated to the so-called fish-eye configuration (i.e. mirror-like circular crack centered at the inclusion). In this nitrided NCI no fish-eye configuration was visible at any stress level.

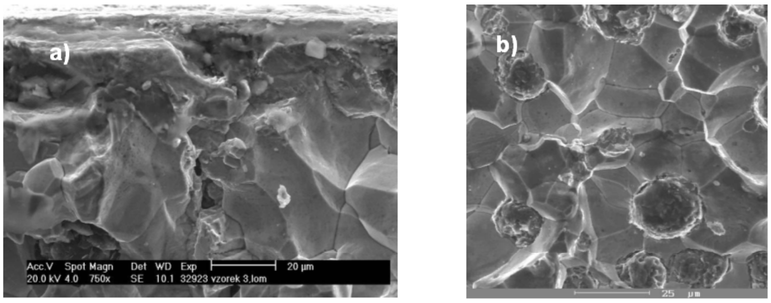

Fig. 4 - Fracture surface of specimen 4 - a) nitrided layer with carbides,
b) intercrystalline cleavage in diffusion zone, SEM

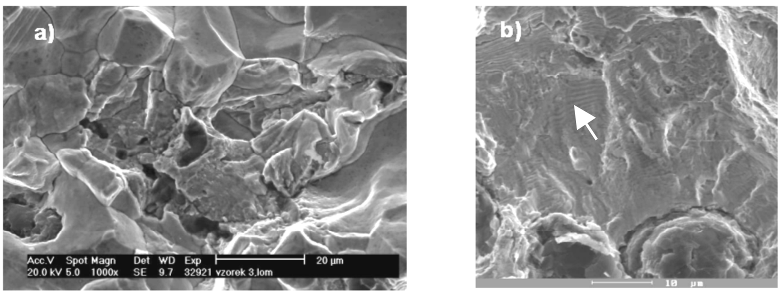

Fig. 5 - Fracture surface of specimen 3 – a) diffusion zone with carbides
b) fatigue region with striations, SEM

From inspection of macrofractograph on SEM it was found that multiple sites of fatigue crack initiation were confirmed by the presence of radial ridges on the fracture surfaces. Cracks initiated at casting defects (micro shrinkages) found below the white layer while no crack initiation occurred at internal graphite particles. Sometimes, carbides and micro shrinkage combined to form a weak place in the white layer where crack could initiate (see Fig. 4a). Initiated cracks then propagated in two directions. Transcrystalline cleavage characterized the growth through the white layer to the surface. The fatigue cracks propagation into the material (from micro shrinkage at the interface of white layer and diffusion zone) was characterized initially by local plastic deformation of ferrite around graphite nodule. Then crack continued in diffusion and sub diffusion zone first predominantly by intercrystalline decohesion along grain boundaries of ferrite grains, see Fig. 4b and 5a and then by formation of fine striations, see Fig. 5b. The fine striations were observed on the fracture surfaces of both specimens but less frequently in the fatigue crack growth region of specimen 4. The presence of striations supports plastic deformation mechanisms of ferrite. In the region of final static failure of both specimens, the crack propagated by transcrystalline ductile fracture of ferrite with dimple morphology.

Fatigue crack paths

A clarification of the different fracture micromechanisms operative in the two specimens with different fatigue life is obtained from inspection and comparison of their fatigue crack paths (or fracture profiles) shown in Fig. 6.

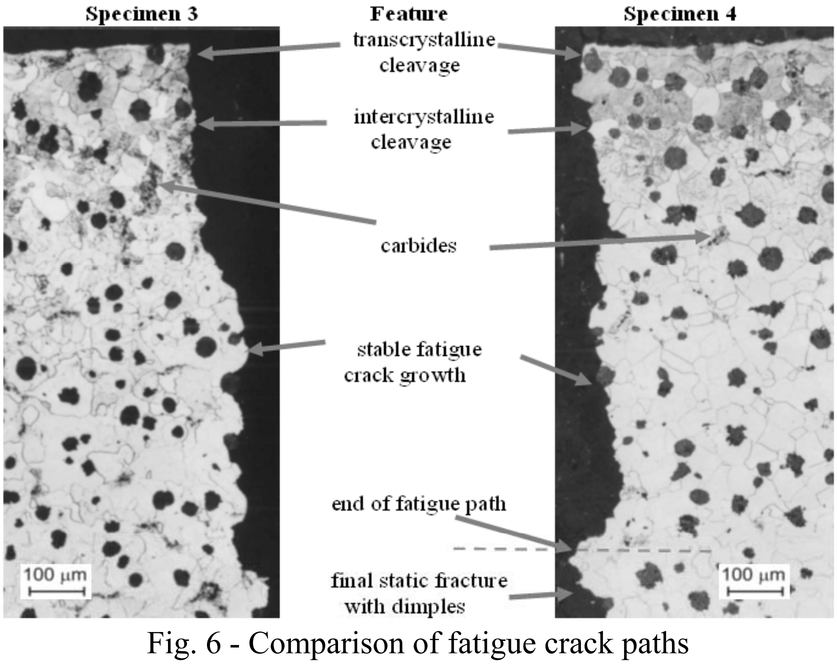

Fig. 6 - Comparison of fatigue crack paths

The pictures show also the material structure obtained by etching. Therefore it is easy to identify the different zones that develop below the surface upon nitriding starting from the white layer, then the diffusion zone in dark gray, the sub-diffusion zone in light gray (compare to Fig. 1) and the base material as a white matrix with a distribution of black graphite nodules. A discontinuous network of carbides is also observed. The first part of crack propagation in the white layer is by transcrystalline growth. Growth in the diffusion zone is then by intercystalline cleavage and it is influenced by nitrides on the ferritic grain boundaries, see Fig. 7a. This mechanism is more extensive and severe in specimen 4 (short life). It is associated to a local high N content (i.e. EDS analysis of the diffusion zone showed that N content decreases more rapidly in specimen 3 compared to the specimen 4, [10]) and high microhardness (i.e. comparison in Fig. 2).

Stable fatigue crack propagation below the white layer is often coupled with striation (see Fig. 5b) and is associated to transcrystalline growth in the sub-diffusion zone. While fatigue crack initiation is not observed at graphite nodules, crack propagation direction through the ferrite matrix is partly influenced by the presence of graphite nodules. A shorter stable crack propagation phase is observed in specimen 4 (see indication of the end of fatigue crack in Fig. 6) compared to specimen 3, coherently with the measured number of cycles to fracture in the two specimens. Final fracture occurs in the base NCI and is always characterized by extensive plastic deformation of ferrite around graphite nodules and dimple formation.

Two other considerations can be advanced in order to discuss differences in fatigue behavior observed in Fig. 3. The first is clarified with the help of Fig. 7 where the magnified pictures of the surface layer of Fig. 1a show the presence of extensive nitride build-up on the ferrite grain boundaries below the white layer, Fig. 7a, and the presence of actual cracks in the white layer, Fig. 7b. Both these features could represent sources of weakness that favor early crack initiation in specimen 4 compared to the sounder surface layer of specimen 3.

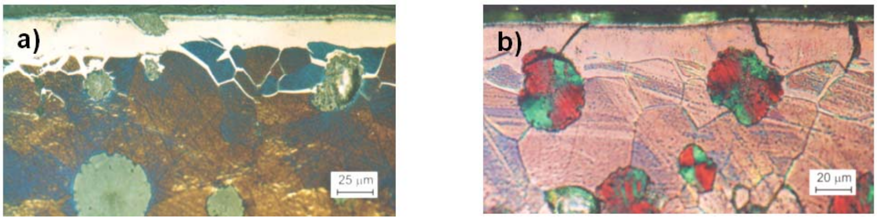

Fig. 7 - Nitrided layer of specimen 4- a) nitrides on the boundaries of ferrite grains, etched with Klemm I [9] b) cracks in white layer, etched with nitric acid, polarized light

The second observation is related to the presence of a higher content of carbides at eutectic cell boundaries in specimen 4 compared to specimen 3. It follows that the material of the original castings from which the specimens were extracted, although nominally the same, could present a slightly different chemical composition. Significant presence of carbides in NCI is not desirable because it weakens the material, [5]. The high content of carbides in specimen 4 influenced the nitriding process and, consequently, the quality of the white layer (porosity, hardness, micro cracks). These factors combine to reduce the fatigue life.

Interpretation for fatigue life of NCI in the presence of nitriding

The scheme of Fig. 8 summarizes i) the current understanding of the parameters involved in controlling fatigue crack initiation and fatigue life of reversed bending fatigue experiments and ii) the material features affecting fatigue life determined in this study and iii) the fatigue crack paths.

Local stresses vary as shown with σ_b the fully reversed stress amplitude. It gradually decreases because it is associated to the bending loading from maximum value on the surface. Residual stress distribution σ_{rs} is not known exactly but based on evidence in nitrided steels the compressive maximum stress (i.e. 200 – 300 MPa) is expected near the surface and goes to zero at the case/core interface. This introduces a positive mean stress effect that decrease with depth from the surface. Finally from the microhardness profile of Fig. 2, the local material strength σ_h is expected to be maximum near the surface (below the white layer) and to decrease to the core (base) strength.

The NCI microstructure is schematically represented as a combination of eutectic cells, each containing a graphite nodule and divided in several ferrite grains. However, two schemes are used to differentiate between two NCI with different content of carbides and micro shrinkages accumulated at eutectic cell boundaries. Upon nitriding, the white layer can be cracked and with grain boundary nitrides (see Fig. 7) in the material model to the left or sound and compact in the material model to the right. Crack initiation can be at micro shrinkages or carbides at eutectic cell boundaries. Early fatigue crack propagation is intercrystalline in the nitrided layer of both materials. Stable crack propagation is shortened in the material with higher content of nitride at ferritic grain boundaries (i.e. specimen 4), because it results in a more brittle fracture behavior. The schemes of two alternative fatigue crack paths based on the results of the fatigue fracture surface investigation are presented in the same Fig. 8.

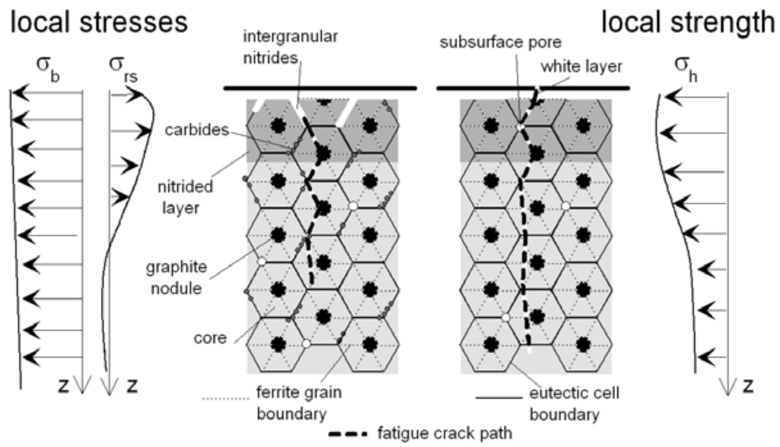

Fig. 8 – Scheme of stress and strength profiles in the nitrided NCI and idealized microstructures and crack paths for short fatigue life (left) and long fatigue life (right)

The fatigue life of a structural part is customarily interpreted in terms of the summation of the life required to initiate a crack and the life spent when the fatigue crack grows stably to final fracture. It appears that the initiation phase can have a dominant effect in the case of and optimized material and nitriding treatment because of the synergistic effect of the compressive residual stress system and the hardened material surface layer. Once a fatigue crack is initiated, however, the sequence of crack growth mechanisms is always the same with a limited influence of their respective extent. To fully exploit the potential of the nitriding treatment, therefore, it is important to control the quality of the base material (i.e. low content of carbides and of micro shrinkage) and the attainment of a steep microhardness profile (i.e. penetration of nitrogen) so that the initiation phase is maximized.

CONCLUSIONS

This study has investigated the influence of a nitriding treatment by the patented Nitreg® Controlled Potential process (Nitrex, USA) on the fatigue response of GJS 400 nodular cast iron and the material and treatment conditions that influence the fatigue life. The following conclusions are reached:

- Tests on smooth specimens of untreated and nitrided nodular cast iron demonstrated a very significant increase in the fatigue limit, but also scatter in the fatigue life data.
- Factors, such as structure of nodular cast iron, penetration depth and concentration of nitrogen on the boundaries of ferritic grains below the white layer, were found to be the causes of the scatter in fatigue lives because they influence the fatigue crack initiation and propagation mechanisms.
- Optimized characteristics of the basic material and of the nitriding treatment result in excellent fatigue performance of nitrided NCI parts.

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