DEFECT CHARACTERIZATION AND FATIGUE OF NITRIZED NODULAR CAST IRON

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Abstract

Nodular cast iron (NCI) is a construction material with a wide range of applications in engineering practice. Since fatigue resistance of NCI is sensitive to surface conditions, the nitriding treatment can be applied because it promotes the formation of a hard, strong surface layer and a system of compressive residual stresses. On the other hand, the fatigue phenomenon is affected by the presence of defects, because they can promote crack initiation. In the case of NCI, the graphite nodules themselves are a first important form of matrix discontinuity. They are homogenous in size (several tens of microns) and spherical in form. Cast materials also contain a population of other defects such as pores and shrinkage cavities that inevitably occur during material solidification. The relative importance of the previously described discontinuities is determined in this paper using metallographic techniques, image analysis software and the statistical Gumbel distribution to describe defect and graphite nodule size populations of specimens after fatigue testing.

This characterization step is then used to discuss the results of fatigue tests performed on nodular cast iron specimens in the untreated and nitrided conditions. Results of fatigue behavior after nitriding show a significant increase in fatigue limit of nodular cast irons. Different trends in the S/N curves are in dependence of structural characteristics.

1. INTRODUCTION

NCI combines the cost-effective casting technology with high fatigue strength [1] and it can be produced according to the classical or synthetic casting procedure, which is more economical because steel scrap is added to the charge instead of a part of pig iron [2]. Until recently ferrosilicon (FeSi) has been mainly used as an additive to the liquid metal to increase the Si content. In the present time, however, silicon carbide (SiC) is predominately used as addition because it increases not only the content of silicon but also the content of carbon [2, 3].

The surface characteristics of NCI may be modified for fatigue-critical applications by thermo-chemical surface treatments, such as nitriding. A surface exposed to a nitriding medium will generally form two distinct layers. The outside layer is called white layer and its thickness generally ranges between zero and 25 µm. Underneath the white layer, there is the diffusion zone and subdiffusion zone [4].

For fatigue-critical application, the population of defects created in the casting process and the graphite nodule characteristics influence NCI fatigue behavior. Typically, fatigue endurance is reduced when the size of porosity increases. Therefore, the quality of the castings is related to the porosity control.
The present work is aimed to identify the influence of different structural factors on fatigue lifetime of nitrided NCI. Light microscopy was performed to analyze the microstructure and pores in the material after fatigue testing. The statistical method proposed by Murakami [5], based on the largest extreme value determination was used to evaluate the porosity size population. Fracture surfaces were selected to determine places of crack initiation and the fracture micromechanisms of nitrided specimens.

2. MATERIAL AND EXPERIMENT METHODOLOGY

As experimental materials were used: i) the classical melt of nodular cast iron (denominated melt B) with ferritic matrix, and ii) 2 synthetic melts of NCI (denominated melt C and T) produced with addition of SiC into the liquid metal with different content of effective ferrite (EF). The chemical composition of melts was similar with approximately eutectic composition, see table 1.

<table>
<thead>
<tr>
<th>MELT</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Cu</th>
<th>Mg</th>
<th>Ni</th>
<th>S_e</th>
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<td>B</td>
<td>3.609</td>
<td>0.131</td>
<td>2.684</td>
<td>0.033</td>
<td>0.009</td>
<td>0.034</td>
<td>0.037</td>
<td>0.047</td>
<td>0.021</td>
<td>1.04</td>
</tr>
<tr>
<td>C</td>
<td>3.610</td>
<td>0.2</td>
<td>2.38</td>
<td>0.03</td>
<td>0.016</td>
<td>0.025</td>
<td>0.204</td>
<td>0.039</td>
<td>-</td>
<td>1.09</td>
</tr>
<tr>
<td>T</td>
<td>3.698</td>
<td>0.220</td>
<td>2.724</td>
<td>0.023</td>
<td>0.011</td>
<td>-</td>
<td>0.124</td>
<td>0.051</td>
<td>-</td>
<td>1.07</td>
</tr>
</tbody>
</table>

Fatigue specimens were extracted from different real castings in the case of melt B and from melted bars (19) of melt C and T. Two sets of smooth fatigue specimens with 6-mm-dia were prepared by machining. Then, one set of specimens of each material was subjected to a nitriding treatment. A Nitreg® patented controlled potential process was used on specimens of melt B and an optimized gas nitriding treatment on specimens of melts C and T.

The fatigue data for untreated and nitrided specimens were obtained on a rotating-bending testing machine operating at 50 Hz (i.e. load ratio R = -1). The fatigue limit \( \sigma_{oc} \) was determined according to a reduced staircase method [4] for melt B. For melts C only two stress amplitude levels were investigated to assess the trend of S/N curves. Different stress amplitude levels were applied on only nitrided specimens of melt T to investigate the S/N curves trend.

The structural analysis was performed on polished and etched specimen cross-sections with the light metallographic microscope according to the standard STN 42 0461 and according to the methods of quantitative metallography.

The image analysis program LUCIA Metallo 5.0 was applied to extensive and detailed measurement of porosity on no etched metallographic sections. To characterize pore size the Largest Extreme Value Determination (LEVD) theory proposed by Murakami [5] was used.

The fatigue fracture surfaces were investigated in SEM on selected nitrided specimens. Nitrided specimens tested at the same stress level and showing different fatigue lives were selected to identify possible sources of weakness in case of both types of material.

3 RESULTS AND DISCUSSION

3.1 Structural characterization

The structure of melt B was characterized by ferritic matrix with a regular distribution of graphite nodules with size ranging from 15 to 60 \( \mu \text{m} \), Fig. 1a. A significant discontinuous network of carbides with microshrinkages on the boundaries of eutectic cells was observed, too. The structure of specimens taken from melt C was characterized by significantly different
content of EF, Fig. 1b, c. The EF content for specimens with almost fully ferritic matrix was from 86 to 70 %, for ferritic-pearlitic matrix from 69 to 52 % and for pearlitic-ferritic matrix from 51 to 41 %. The graphite nodules were observed in fully or not fully globular shape predominately with size ranging from 30 to 60 µm and with small ratio of size ranging from 60 to 120 µm. The structure of melt T showed the similar trend and groups characterized by a different content of effective ferrite, Fig. 1d-f.

Specimens were divided according to the EF content into three groups, too: i) nearly ferritic matrix with EF in the range from 86 to 78 %, ii) high ferrite content with EF from 76 to 71 % and iii) ferritic-pearlitic matrix with the lowest EF content ranging from 65 to 62 %.

A nitrided layer of classical and synthetic melts was formed by a thin white layer (WL) on the surface of specimens, diffusion zone (DZ) and subdiffusion zone (SDZ), Fig. 2a.

The white layer was continuous with variable thickness from 10 to 28 µm for melt B, respectively from 9 to 33 µm for melt C, and for melt T from 5 to 25 µm in the dependence on the presence of graphite particles.
A thicker white layer and diffusion zone were identified in areas where graphite particles were present. In all specimens a thin dark layer, which is most probably a carbonitrided layer, in white layer was identified, when a high magnification was used, Fig. 2b.

3.1 Fatigue properties

The fatigue data obtained on the different melts and in the as-cast and after nitriding are now presented and discussed. In all cases the nitriding treatment is demonstrated to give a very significant improvement of the fatigue response, comparable of that observed in steels [3]. The increase in the fatigue strength associated to the nitriding treatment is due to the simultaneous formation of the hardened surface layer and of favorable compressive residual stresses.

The S/N curves of the untreated and nitrided melt B are shown in Fig 3a. The fatigue limit is $\sigma_{oc} = 169$ MPa for untreated and $\sigma_{oc} = 381$ MPa for nitrided NCI. The fatigue life data of the nitrided NCI are fitted with two parallel S/N curves because specimens subjected to the same applied stress amplitude showed fatigue lives differing by more than two orders of magnitude, an indication of different initiation mechanisms described elsewhere, [6]. The data and the trend of S/N curves of melt C (Fig. 3b) showed higher number of cycles to failure for the same applied stress amplitude for untreated melt C because of the lower EF. The fatigue data of untreated and nitrided specimens of melt C showed no significant dependence of number of cycles to the failure on content of EF within individual specimens. The data of melt T are only for the nitrided conditions and are quite scattered about the mean S/N curve shown in Fig. 3c. A direct comparison of the response of the different melts is presented in the plot of Fig. 3d where data points are eliminated and only the trend lines are shown. In the untreated condition, the reduction of the effective ferrite in the matrix tends to increase the fatigue resistance. This increase is not maintained after nitriding. All nitrided data appear to

Fig. 3 Fatigue data
distribute within a single scatter band. However, a difference in response appears not in terms of absolute stress levels rather on the slope of the S/N curves. This could be an indication that different micromechanisms are operative in the pearlite and ferrite after ntriding. This point, however, needs further verification.

3.2 LEVD application

An extensive investigation was performed to find the influence of different structural factors on fatigue lifetime. The most important factors influencing fatigue life of NCI are the

Fig. 4 Defect size dependence of selected specimens
content of EF, number of graphite particles per mm$^2$ and size and shape of casting defects (microshrinkages). Many microshrinkages in the matrix were found in the structure of NCIs. In the case of synthetic melts the microshrinkages were mainly found in pearlitic areas.

The Murakami method for the determination of the largest extreme value distribution (LEVD) was applied to characterize casting pores. The measured data using LEVD method with function

$$y_j = f(\sqrt{\text{area}_{\text{max},j}})$$  \hspace{1cm} (1)

were plotted to the graph shown in Fig. 4, where a linear trend is observed.

The largest defect size was evaluated on specimens of each melt tested at the same stress amplitude level and with a very different fatigue life. The specimen B3 with long fatigue life, Fig. 2a, is characterized by smaller pore sizes, Fig. 4a, than the specimen B4 with short $N_f$. The graphite nodules size shown in the same plot is almost similar for both specimens of the melt B.

The pore sizes and fatigue lives show similar results in the case of synthetic C and T melts. Specimens C4, C17 and T19, T13 represent results for two specimens of each group tested at the same stress amplitude. The specimen C4 with lower content of EF (EF = 68 %, $N = 112$ mm$^2$) with high fatigue life ($N_f = 3 163 358$) is characterized by small pore size, Fig. 4b, compared to the specimen C17 with high content of EF (85 %) and comparable nodule count ($N = 110$ mm$^2$) with low fatigue life ($N_f = 359 524$), Fig. 2b. The graphite nodules size is nearly similar for both specimens of the melt C. The specimen T19 (EF = 81 % and $N = 127$ mm$^2$) with smaller pore size, Fig. 4c, showed high cycles to the failure compared to the specimen T13 with low fatigue life, Fig. 2c, which shows higher content of EF (78 %) bad small content of nodule count ($N = 91$ mm$^2$). A contribution to the short fatigue life may be associated to the larger size of graphite nodules for specimen T13, Fig. 4c.

### 3.3 Fractographic analysis

A macrograph of fatigue fracture surface of nitrided NCI specimens showed two specific areas: i) light fatigue region on peripheral area of fracture surface and ii) dark region situated in central part of final static fracture, Fig. 5. The extent of the fatigue areas increased with decreasing of stress amplitudes.

![Fig. 5 Macrograph of fatigue fracture surface, SEM](image)

![Fig. 6 Radial stairs, SEM](image)

Multiple initiations of fatigue crack, Fig. 6, were associated to the presence of radial stairs on the fracture surface of both (classical and synthetic) types of nodular cast irons in untreated and nitrided conditions. The cracks were initiated from different places: i) the white
layer with small microcracks, ii) interface of the graphite particles and discontinuous white layer, or iii) cast defects situated below the white layer, Fig. 7a.

![Fig. 7 Fatigue fracture surface, SEM](image)

The small microcracks were found on the metallographic section in case very high hardness of white layer, which has to be continuous, hard and elastic.

Fatigue crack propagation in nitrided layer was characterized by transcrystalline cleavage and by intercrystalline decohesion along grain boundaries in diffusion and sub-diffusion zone, Fig. 7b.

The fatigue region is characterized by presence of striations, Fig. 7c, typical for ductile materials. A transcrystalline ductile fracture with dimple morphology in ferrite and with fine dimple morphology in pearlite in case of its presence in the metal matrix, were identified in final static fracture, Fig. 7d.

4. CONCLUSION
The following main conclusions are reached:

- the amount of effective ferrite and number of graphite particles do not show direct influence on fatigue behavior,
- the nitrided layer with the different thickness was not continuously covering the graphite particles,
fatigue cracks initiated from many places i.e. the graphite particles on surface, microshrinkage under the white layer, pore size with other structure characteristics influence the fatigue lifetime.

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REFERENCES