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Regular article White etching matter promoted by intergranular embrittlement

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article info abstract

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To determine the role of brittleness in producing "white etching matter," rolling elements were intentionally case-carburize heat treated to produce an embrittled case. The intergranular embrittlement was confirmed by characterizing the fracture surfaces of the steel. Post rolling contact fatigue investigations showed subsurface cracks, forming preferentially along the grain boundaries, and white etching matter was determined to be present along these intergranular cracks. The white etching matter was characterized by electron microscopy. These results provide novel insights into cause-effect relationships and evolution mechanisms associated with white etching matter.

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The microstructural soundness of a material plays a key role in the durability and robustness (resisting damage under severe loads) of a mechanical component that is made of the material. Bearings are well known mechanical components used in motion and load transmission. Because of their vital role in running the machinery, bearings are designed to deliver required life with very high reliability. Therefore, to suit the application, the steels used to manufacture the bearings are well controlled in terms of their cleanness, composition and microstructure [\[1\]](#page--1-0). Different heat treatment processes are used in bearing manufacture, including through hardening, austempering, case carburizing and induction hardening [\[1\]](#page--1-0). The appropriate heat treatment process is selected based on the bearing's steel type, bearing size, application and cost requirements.

Because of their widespread usage, the damage modes associated with bearings are well documented in literature [\[2\].](#page--1-0) Some of these damage modes are generated by rolling contact fatigue (RCF), and they involve microstructural changes within the bearing steel [\[3,4,5,6\].](#page--1-0) Metallographical examination of the microstructural changes by the application of Nital (etchant) reveals damage to the steel which, based on its appearance, is referred to as "dark etching" (deeply etched) or "white etching" (not etched) areas [\[1,4\].](#page--1-0)

This microstructural alteration is generally observed in the subsurface regions of bearing components, because that is where cyclic shear stresses act during the operation of a loaded bearing. Although the dark etching areas and white etching areas are well characterized by different microscopy techniques, the cause-effect relationships associated with these areas are still under debate [\[7,8\]](#page--1-0). In this context, white

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<http://dx.doi.org/10.1016/j.scriptamat.2016.11.030> 1359-6462/© 2016 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved. etching areas have drawn intense interest in recent years because they are often found in bearings with premature failures [\[9,10\].](#page--1-0) However, it is not completely clear whether the white etching areas are the root cause of failure or they are the consequence of some other phenomenon in the steel [\[11\].](#page--1-0) If white etching areas are present along the cracks, the literature often describes them as "white etching cracks" [\[12\].](#page--1-0) Since the white etching areas are also present in other locations for example, at inclusion sites (in the morphology of butterflies) [\[13\]](#page--1-0) in this paper, we use the common term, "white etching matter" (WEM), as denoted in recent publications [\[11\].](#page--1-0)

As previously mentioned, the premature failure of bearings is often attributed to white etching matter (or white etch cracking) [\[8,9\].](#page--1-0) Multiple mechanisms have been proposed for the formation of WEM and how it leads to functional damage of the bearing. External factors are often cited as the root causes for the WEM, including i) hydrogen diffusion into steel from lubricating oil [\[14\]](#page--1-0); ii) very high impact loads on bearings that cause adiabatic shear bands in the steel [\[15\]](#page--1-0); iii) negative slip and high slide-to-roll ratios [\[16\]](#page--1-0); and iv) improper bearing fitting and operation [\[17\]](#page--1-0). Some reports suggest that WEM generated at inclusion sites (butterflies) may interconnect and cause crack networks in bearings [\[18\]](#page--1-0).

Critical assessment of these reports suggests [\[11\]](#page--1-0) that since the WEM is always associated with cracks or some other weak areas (inclusions) in steel, the generation of WEM may be related to brittleness (or mechanically weak areas in microstructure) of the steel [\[11\].](#page--1-0) Tougher microstructures, such as carbide-free nano-bainitic structures, have been proposed as a means of mitigating the problem [\[11\]](#page--1-0). More experimental and computational evidence is needed to understand how brittleness and the material microstructure play a role in generating WEM and to prevent those premature bearing failures that are believed to be caused by WEM.

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To determine the role of steel's brittleness, in this report the investigators intentionally induced brittleness into steel samples (rolling elements) and subjected them to RCF conditions. A variant of a case carburizing heat treatment was selected to induce the brittleness. Case carburized steel is known to undergo intergranular embrittlement and possesses coarse martensitic microstructure [\[19,20\]](#page--1-0) when the carburizing is conducted at elevated temperatures ($>$ 950 °C) with high carbon potentials $(>1$ wt% C) followed by a direct quench and a lowtemperature $(-150 \degree C)$ tempering step. Because of the intergranular embrittlement, overload fractures in the steel tested showed intergranular fracture features in the carburized case [19–[21\];](#page--1-0) in other words, fractures occurred along the grain boundaries. This intergranular embrittlement can be mitigated by refining the case microstructure. The refined microstructure promotes transgranular fracture surfaces and significantly improved fatigue performance [\[19](#page--1-0)–20]. This type of microstructure has been proposed to resist the WEM formation and premature bearing failures in wind turbine applications [\[22\]](#page--1-0).

Because of their functional requirements, case carburized bearings generally contain refined microstructures [\[1\].](#page--1-0) For easier understanding, examples of coarse and refined martensitic microstructures near the surface (case) of carburized steels and respective overload fracture morphologies are shown in the supplementary information, Fig. S1. In this study, to obtain the intergranular embrittlement, rolling elements were intentionally carburized at 1000 °C, followed by a direct quench in oil and a low-temperature (150 °C) tempering step. The carbon potential during the case carburizing treatment was adjusted to have a carbon content of 0.8 wt% in the surface of finished rolling elements. For the rolling elements, we used a modified AISI 8620 grade steel with a composition (wt%) of C: 0.2, Mn: 0.83, P: 0.013, S: 0.014, Si: 0.34, Cr: 0.57, Ni: 0.27, Mo: 0.1, Cu: 0.16, and Al: 0.037. Materials were characterized by optical microscopy and a scanning electron microscope (SEM). The SEM used in this study is the FEI Versa 3D Dual Beam SEM (electron and ion beams), equipped with focused ion beam (FIB) milling capability. Microhardness across the case region is measured using CLARK microhardness tester with a load of 500 gm. Retained austenite and residual stress were measured using a PROTO LXRD system.

Fig. 1a shows a typical optical micrograph of the carburized roller displayed in the case region; this micrograph shows coarse tempered martensite (darker areas) in a retained austenite matrix (bright areas). X-ray diffraction (XRD) measurements showed about 17% of the retained austenite in the case region and a significant compressive residual stress of −440 MPa at the surface. The micro-hardness profile, as measured across the case region, is shown in Fig. 1b. The plot shows HV (Vickers Hardness) numbers and equivalent HRC (Rockwell Hardness, scale C) numbers on the "Y" axes. High hardness at the surface region and a gradual decrease toward the core region is a typical characteristic of case carburized steel, caused by the high carbon levels diffused into the steel in the near-surface region (case) and the subsequent quench hardening step. To determine the roller's brittleness, a notch was made in the roller and it was fractured by impact loading (fractured by overload) at room temperature. The fracture surfaces were examined with scanning electron microscopy (SEM).

Fig. 1c, d and e shows images of the fracture surfaces. Fig. 1c shows a macro photograph of the fracture, where a clear difference between the appearance of the case and core regions can be seen. The case is observed to possess rough and bright features, whereas the core shows a smoother morphology. The rough and bright features of the case are characteristic of brittle fracture. An SEM image of the fracture surface from the case region is shown in Fig. 1d. The fracture surface shows smooth morphologies of the grains, which in fact suggests that the fracture took place along the grain boundaries. No ductile fracture features, such as dimples and voids, are observed in the case region. These fracture morphologies clearly suggest an intergranular brittle fracture in the case region.

Because of the high temperature used in the carburizing treatment and the consequent grain growth phenomenon, grain sizes in the case region (Fig. 1d) vary between 20 and 60 μm. The coarse martensite shown in Fig. 1a is also a consequence of this high-temperature carburizing followed by a direct quench step. The fundamental reason for the intergranular embrittlement was showed to be related to segregation of impurities and carbon to the prior austenite grain boundaries [\[19,20\]](#page--1-0). The fracture surface from the core region, Fig. 1e, has dimple and void-like morphologies all over its surface, indicating the ductile nature of the core. Since the core has a low carbon content, the core did not undergo the embrittlement phenomena. Therefore, the core showed features of ductile fracturing.

To understand the damage mechanisms associated with these rollers in rolling contact fatigue (RCF) loading conditions, the rollers were assembled into the test rig and subjected to a RCF load [\[23\]](#page--1-0). As described in ref. [\[23\],](#page--1-0) in this type of testing, four assemblies at a time are tested in a test rig under the same load. The test is conducted until

Fig. 1. Characterization of steel after the case carburizing. (a) Optical metallograph taken from case region of carburized steel. (b) Micro-hardness distribution across the case region. (c) Photograph showing the overload fracture of the case carburized steel. (d) and (e) respectively show SEM images from the case and core regions of the fracture surface shown in (c).

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