

Fatigue Properties of Carburized Extra-fine Ni Steels

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Abstract

High compressibility Ni-Mo steels are leading candidate materials for high density PM steel gears. Recently developed admixed Ni-Mo steels containing extra-fine Ni powder limit the formation of soft Ni-rich austenitic phases thought to be detrimental for contact fatigue performance of PM steels. FLN2-4405 mixes containing both standard (STD Ni) and extra-fine Ni (XF Ni) powder were compacted into Charpy bars and sintered to nominal $7.05 - 7.25 \text{ g/cm}^3$ density at 1120 to 1180 °C in an industrial sintering furnace. Charpy bars were machined into RBF bars and then case carburized. Rotating bending fatigue tests were carried out and the results were compared. Microstructures obtained for the carburized steels exhibited some differences depending on the type of admixed Ni powder. Case microstructures were similar for both steels, consisting of acicular martensite, retained austenite and Ni-rich areas. Core microstructures were bainitic with Ni-rich areas in STD Ni steels. XF Ni steels had a mixture of bainite and martensite in the core with few Ni-rich areas, indicative of higher hardenability. Contrary to expectations, the bending fatigue endurance limit was approximately 10% higher for standard admixed Ni steels compared to extra-fine Ni steels. Crack initiation occurred in surface porosity, with crack propagation occurring by both transgranular fracture and ductile overload. Improved fatigue performance could be anticipated with a reduction in surface porosity and optimization of the steel composition and carburizing cycle to take advantage of the increased hardenability of the XF Ni steels.

Introduction

Gear performance strongly depends on the microstructure of steel and the presence of microstructural features that either decrease strength or raise the locally applied stress [1]. Carburizing the steel gear to achieve a high carbon hard case and low carbon ductile core achieves favourable residual compressive stresses during quenching, with the resultant material exhibiting high fatigue resistance. The case microstructure should consist of martensite with retained austenite and no non-martensitic transformation products [2]. For powder metallurgy (PM) gears, control of the microstructure includes the amount, distribution and shape of residual porosity.

Achieving high density is one of the key PM material requirements, as the presence of pores severely limits fatigue performance. In order to achieve high density in complex geometry PM steel parts for which PM can be competitive with machined wrought steels, high compressibility Fe powders are needed. This requirement leads to the preference by PM parts makers of Fe powders with low prealloy content, in particular Ni and other ferrite hardening elements such as Cr and Mn. On the other hand the steel must have

sufficient hardenability once the part has been formed, which runs counter to the need for high compressibility powders.

Ni is often added to wrought gear steels to improve the toughness of martensite. In PM steels, Ni not only adds toughness, strength, and hardenability to the steel, but also increases sinter density. However the slow diffusion of Ni during conventional sintering typically results in a microstructure consisting of Ni-rich austenitic steel phases. The presence of these Ni-rich areas has traditionally been thought to be beneficial in fatigue performance of PM steels. However more recently, these phases have been shown to be undesirable in contact fatigue performance of surface densified FLN2-4405 steel [3]. Jandeska et al concluded that residual porosity associated with Ni-rich phases was found to act as sites for crack initiation and thought to be responsible for the high scatter in fatigue life.

The work by Jandeska et al lead to an initial rolling contact fatigue (RCF) study on FLN2-4405 steels containing XF Ni powder in order to reduce the size and amount of Ni-rich phases [4]. Following the same processing conditions as the previous work by Jandeska, steels were high temperature sintered (1260 °C) to nominal 7.4 g/cm³ density, machined, surface densified by roll compaction and carburized. RCF results on a ZF bench tester indicated that XF Ni steels had superior performance to baseline 8620 wrought steel at 1900 MPa contact pressure and at 2500 MPa pressure approximately twice the life of the STD Ni steel processed under similar conditions. The microstructure of the XF Ni steel revealed complete dissolution of Ni and suggested that lower processing temperatures and alloy content could be used to achieve the desired microstructure.

Early work by the author showed that finer Ni powder improved the bending fatigue performance of nominal 7.0 g/cm³ density FN-0208 steels by approximately 20% in the as-sintered condition [5]. In the heat-treated condition, there was no difference in the estimated endurance limit between FLN2-4405 steels containing standard and XF Ni. The result was explained by the controlling behaviour of the high level of porosity and the martensitic microstructure in the heat-treated condition afforded by 0.85% Mo.

Several bending fatigue studies have been conducted on Ni-containing PM steels. In four point bending fatigue tests, Alzati suggested that Ni-rich phases are beneficial in Distaloy HP-1 and AE when surrounded by a largely martensitic Cu-rich matrix [6]. On the other hand Nigarura et al described the failure mechanism of modified FLN2-4405 as ductile fracture with void coalescence, which was attributed to the presence of soft Ni-rich phases [7]. Bernier et al measured a 5% increase in bending fatigue endurance limit for sinter-hardened steel FLNC-4205 containing 1.5% Cu and 0.5% admixed XF Ni [8]

Chawla showed in single notch edge axial fatigue tests that the crack propagation rate was faster through Ni-rich regions of a pearlitic / bainitic microstructure of as-sintered FLN2-4405 [9]. In a subsequent paper, in comparing prealloyed vs. admixed Ni steels Chawla demonstrated that prealloyed Ni steels have higher fatigue strength than admixed Ni steels [10].

The goal of the current study was to characterize the bending fatigue behaviour of carburized FLN2-4405 steels processed at mid-level densities in the range 7.05 to 7.25 g/cm³. This work was intended as an extension of the promising RCF results on XF Ni steels, in order to more fully measure the fatigue performance of these materials for PM steel gear applications. In addition, the bending fatigue study forms part of a preliminary investigation to support the current “Single Press to Full Density” initiative sponsored by the Center for Powder Metallurgy Technology, in which XF Ni steels are an integral part of the materials test matrix.

Experimental

Sample Preparation

Prealloyed Fe-Mo powder (Atomet 4001 = 0.85% prealloyed Mo, 0.15% Mn) was admixed with 2% Ni (Inco ® Type 123 PM or T110 D), 0.35% graphite (SW 1651) 0.5% lubricant (Kenolube). The powders were mixed in a V-cone blender for 25 minutes without intensification. Charpy bars were compacted at 550 to 830 MPa (40 to 60 tsi) using a 150 ton Gasbarre mechanical press. The steady state die temperature at 830 MPa compaction pressure was approximately 60 C, which was thought sufficient to allow nominal 7.35 g/cm³ green density. However measurement of the sinter density of tested samples revealed an average density closer to 7.2 g/cm³. A total of 150 bars were made per mix for each density. The bars were sintered in an industrial furnace at both 1120 and 1180 °C in a 90/10 N₂/H₂ atmosphere for 45 minutes in the hot zone. Of the sintered steel bars, 25 samples were chosen for machining into RBF test bars according to MPIF Standard 56 [11]. The machined bars were then conventionally carburized using an endo gas atmosphere with carbon potential of 1.1%. The total carburizing cycle time was 2 h 15 min temperature 1600 (equalize) – 1750 F (boost / diffuse).

A total of 18 specimens were tested in rotating bending fatigue tests to evaluate the S-N curve for each steel using the JSME 002 Standard combined with determination of the fatigue endurance limit by the staircase method following MPIF Standard 56 [11,12].

Results

Flow of the mixes containing the two Ni powders was similar (28 s). Apparent density was slightly lower for the XF Ni blend: 3.14 vs. 3.18 g/cm³ for the STD Ni blend. Average combined carbon content in the sintered Charpy bars ranged from 0.25 – 0.26%. Apparent hardness of the as-sintered steels sintered at 1120 °C was 50 and 53 HRA for STD Ni and XF Ni steels respectively. Steels sintered at 1180 C had similar apparent hardness (53 HRA).

Ni distribution was checked in sintered steels prior to machining into RBF bars. Uniformity of Ni distribution was clearly improved in XF Ni steels relative to STD Ni steels. Although a few small Ni agglomerates were present in the XF Ni steels, the distribution of Ni was considered representative of admixed XF Ni steels blended using

conventional mixing equipment. As-sintered microstructures of the low carbon FLN2-4405 steels consisted of ferrite, pearlite and small areas of martensite associated with the edges of Ni-rich regions. The Ni-rich phases were larger and more numerous in the STD Ni steels.

Porosity distribution was measured by both Visilog and checked by Clemex automatic analyses. Pore size distribution most strongly depended on sinter density. XF Ni steels had a greater number of fine pores ($< 2 \mu\text{m}$) and STD Ni steels had more large pores ($>20 \mu\text{m}$) at 7.05 g/cm^3 density. As the sinter density increased, the porosity distribution looked similar for the two Ni steels. Pore shape factor followed a similar trend to pore size distribution and did not reveal a strong dependence on Ni powder type nor on the sintering temperature.

Sintered Charpy bars experienced growth in the long dimension whereas the short cross section dimensions underwent shrinkage from die size for all three sinter densities. Growth was typically greater for the XF Ni steels compared to STD Ni steels, which is the opposite of what would normally be expected for increased Ni diffusion. Some evidence has been found of lamination cracks in the outer regions of the Charpy bars compacted at 840 MPa (60 tsi), however this does not explain the growth behaviour of the lower density steels unless microcracks are more prevalent in the Charpy bars than we suspect at the time of writing. No evidence was seen of blistering or any other distortion of the surface of the sintered Charpy bars.

Apparent hardness of carburized RBF bars was 46 – 48 HRC in the case and 39-41 in the core, with the higher values corresponding to XF Ni steels. Ultimate tensile strength measured on RBF bars was 1350 and 1390 MPa for STD Ni and XF Ni steels respectively. This result compares well with MPIF standard data for quenched and temper FLN2-4405; 1450 MPa at nominal 7.3 g/cm^3 density.

Distribution of Ni-rich phases can clearly be seen in the low magnification pictures of carburized RBF samples in Figure 1. Again the Ni-rich phases are larger and more numerous in the STD Ni steel.

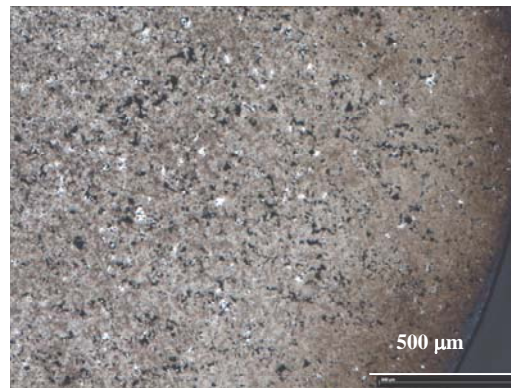
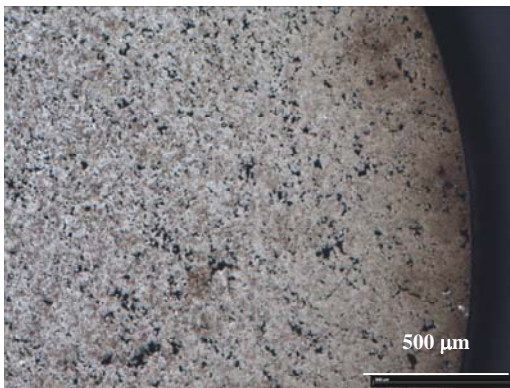


Figure 1. Case carburized steels containing admixed XF Ni (left) and STD Ni powder (right). NiRAs (bright light etching) are more pronounced in STD Ni steel. Magnification 50 X.

The case microstructure of carburized steels consisted of acicular martensite and retained austenite, with Ni-rich areas visible particularly in the STD Ni steels. The core microstructure was largely bainitic with Ni-rich phases, with varying amounts of lath martensite particularly in the XF Ni steels, indicating increased hardenability (Fig. 2). Sintering at 1180 °C visually appears to have improved the pore morphology and to have increased Ni diffusion.

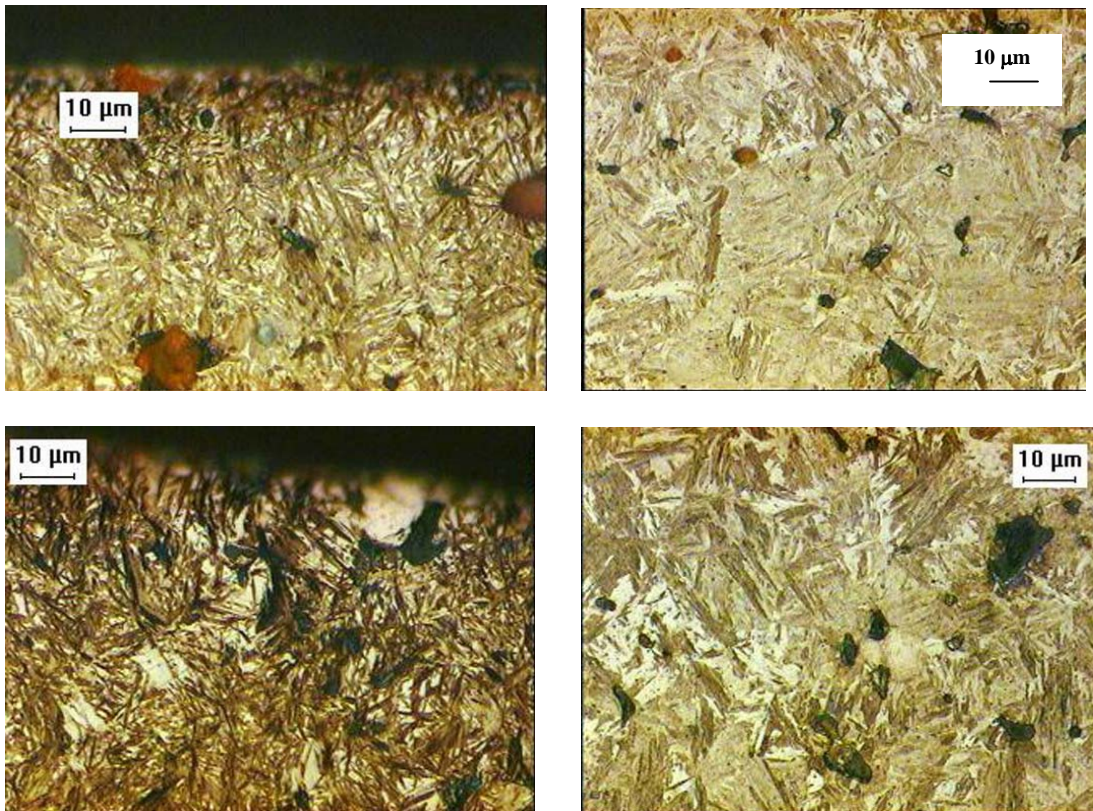


Figure 2. Steels sintered at 1180 °C, case (left), core (right). XF Ni (top) STD Ni (bottom). Magnification 800 X (2% Nital etch).

Microindentation hardness profiles were generated for the carburized steels using 100 gf load. The microindentation hardness ranged from over 800 HV 100 at the surface to 500 – 700 in the core. The profiles were similar for XF Ni and STD Ni steels, with better uniformity in XF Ni steels as a result fewer and smaller Ni-rich phases. Based on the microindentation hardness profile, the case depth was estimated to be approximately 0.5 mm.

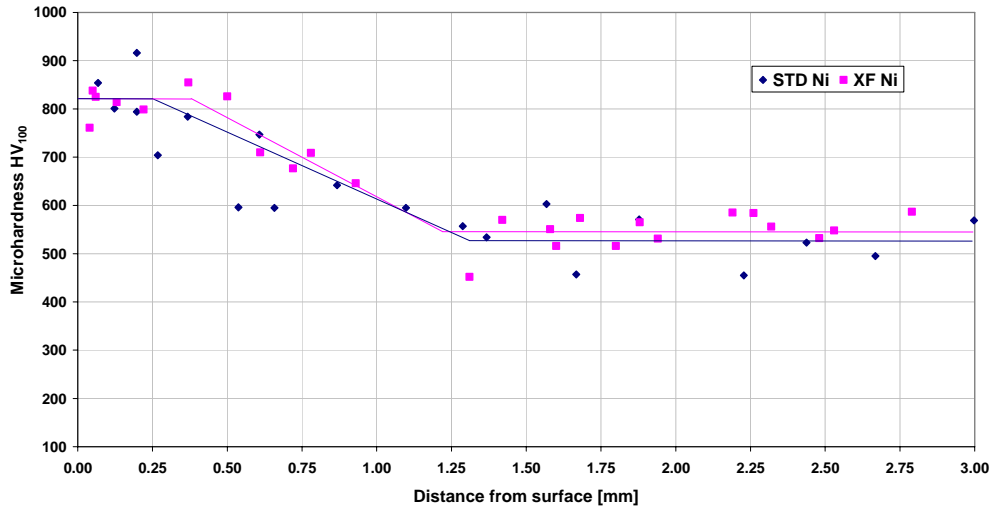


Figure 2. Microhardness profile of carburized steels. Case depth was estimated to be approximately 0.5 mm. Average core microhardness XF Ni was about 50 HV higher than STD Ni steels.

Unfortunately due to numerous unforeseen delays associated with sample preparation and testing of the carburizing steels, only the nominal 7.25 g/cm^3 sinter density results for this study are available at the time of writing. Distortion of the RBF bars during heat treatment made it difficult to meet the concentricity requirements of MPIF Standard 56. Final machining / grinding was not performed on the test bars after carburizing. The average concentricity for the samples was as follows:

$$\text{XF Ni} = 0.0035 \pm 0.0017$$

$$\text{STD Ni} = 0.0039 \pm 0.0018$$

While the lack of concentricity of the carburized RBF samples was up to 5 times the limit recommended by Standard 56, the fatigue data showed no dependence of fatigue life on concentricity of the samples. Although the fatigue endurance limit measured in these tests would be lower than expected for less distorted samples, the data are considered to be valid in comparing fatigue life behaviour between the two steels.

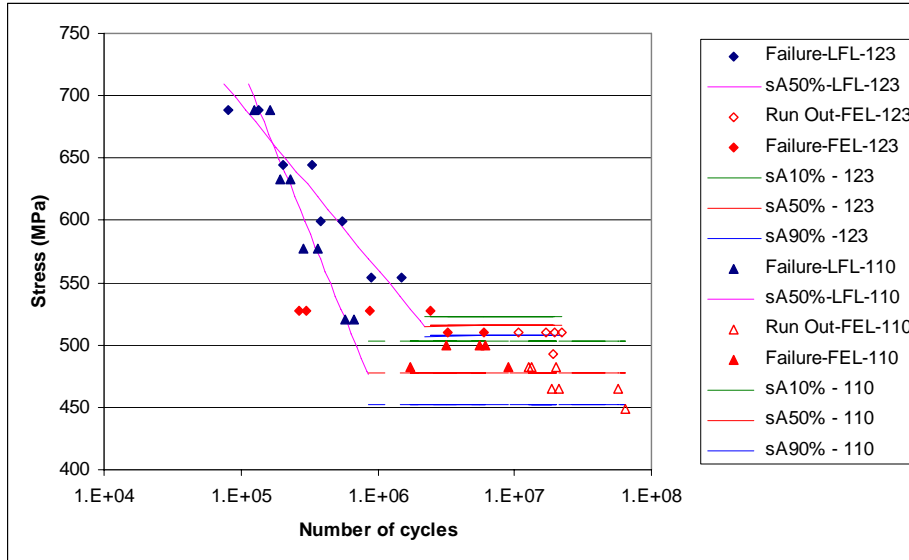


Figure 3 RBF results nominal 7.25 g/cm³ steels.

The rotating bend tests in Figure 3 indicate a statistically significant higher fatigue endurance limit for the STD Ni compared to XF Ni steels. The reason for the difference in slope of S-N curves is most likely related to the slightly more brittle behaviour of the XF Ni steels. Scatter is also notably higher for XF Ni steels.

Fracture surfaces of both steels were similar (Fig. 4). Failure initiated at pores on the surface of the test bars. Fracture in the brittle case was transgranular, followed by a mixture of quasi-cleavage and ductile rupture as the crack propagated into the more ductile bainitic core. XF Ni steels have martensite in core leading to more transgranular (faster) fracture. Crack initiation in porosity on surface same for both steels. Crack propagation faster in less ductile material explains lower fatigue performance of T110 steels

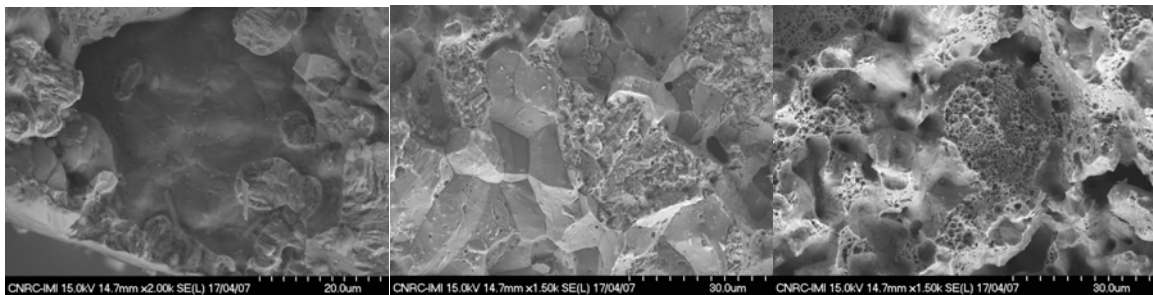


Figure 4. Fracture surfaces of STD Ni steel. Crack initiation at pore (left), intergranular cleavage in the case layer (middle) followed by ductile tearing in the core (right).

Discussion

Need balance between RCF and bending fatigue performance

At PM2Tech-2005 in Montreal, Fillari et al presented bending fatigue results on case carburized FLN-4405 steels with variable carburizing cycle and Ni content [13,14]. The base case carburizing cycle was longer than that employed in this study (180 vs 135 minutes) and the maximum temperature a little lower (1700 vs. 1750 boost / 1600 equalize in this study). Apparent hardness in the as-sintered steels was similar to this study (50 HRA). However apparent hardness obtained by Fillari was much lower for carburized steels, 21 and 40 HRC compared to the results in this study of approximately 40 and 48 HRC for the core and case respectively. For nominal 7.2 g/cm^3 sinter density, the 90% survival limit (σ_{90}) was determined to be 340 MPa, significantly lower than the 450 – 510 MPa obtained in this study. Ultimate tensile strength of $7.25 - 7.4 \text{ g/cm}^3$ sinter density carburized steels ranged from 980 to 1070 MPa, again lower than 1350 – 1390 obtained for STD Ni and XF Ni steels respectively in this study. However unpublished work suggests that the fatigue endurance limit that could be expected for carburized FLN2-4405 steels is closer to 600 MPa [15].

The ratio of fatigue endurance limit : UTS ranged from 0.32 to 0.38 for this study, in line with Fillari's result of 0.34. The RBF data for case carburized steels measured in this study and from Fillari are compared in Figure 4 with standard RBF fatigue data in MPIF Standard 35 [16]. The data from this study look reasonable compared to standard and previously published data.

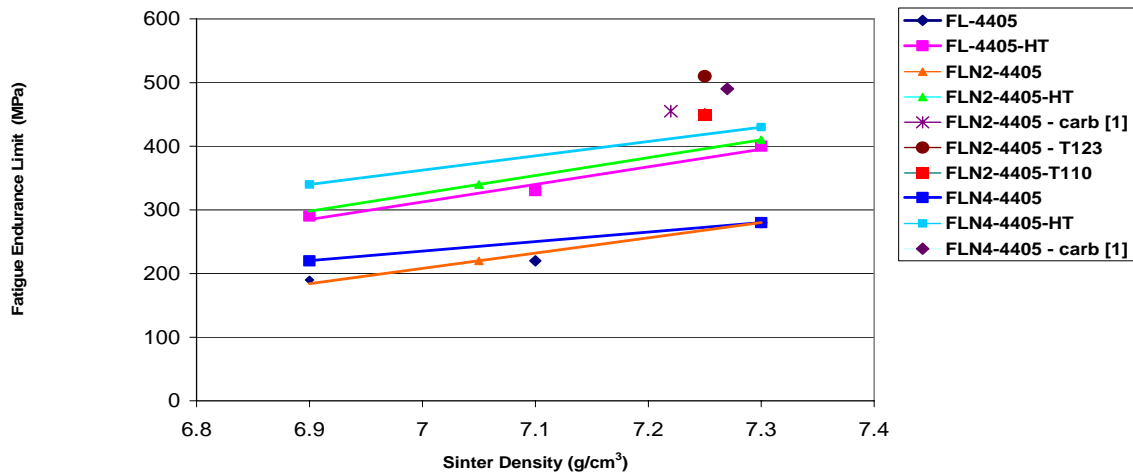


Figure 4. Comparison of RBF data FLN-4405 steels. Trendline reference data MPIF Std. 35, ref [13,14]

The results of this study indicate that crack initiation occurred at porosity on the surface of the test bars. Once the crack was initiated, propagation was relatively fast and had

only a small dependence on the type of admixed Ni powder. XF Ni steels had less resistance to crack propagation due to the higher proportion of martensite in core. These results show that steels processed in same manner with different Ni powder can produce different core microstructures. The fatigue endurance limit of these carburized FLN2-4405 steels could be significantly improved with optimization of the steel microstructure and carburizing cycle in order to take advantage of the greater hardenability of XF Ni steels.

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References

1. J. Wise, D. Matlock, G. Krauss, "Bending Fatigue of Carburized Steels", Heat Treating Progress, Aug./Sept., pp. 33-41 (2001).
2. D. Matlock, K. Alogab, M. Richards, J. Speer, "Surface Processing to Improve the Fatigue Resistance of Advances Bar Steels for Automotive Applications", Materials Research, Vol. 8, No. 4, 453-459, (2005)
3. W. Jandeska, R. Slattery, F. Hanejko, A. Rawlings, P. King, "Rolling Contact Fatigue Performance Contrasting Surface Densified, Powder Forged and Wrought Materials", Advances in Powder Metallurgy & Particulate Materials – 2005, Montreal, pp. 44-55, (2005)
4. T. Stephenson, M. Korotkin, T. Singh, G. Hoffmann, "Performance Improvements in Surface-Densified FLN2-4405 Gears made with Extra-fine Ni Powder", Advances in Powder Metallurgy & Particulate Materials – 2005, MPIF, Montreal, pp (2005)
5. T. Stephenson, T. Singh, S. Sun, Z. Wang, "Effect of Ni Powder Morphology on the Fatigue Properties of PM Ni Steels", Advances in Powder Metallurgy & Particulate Materials – 2002, MPIF, Orlando, pp. 5-153 – 5-164 (2002)
6. L. Alzati, A. Bergmark, J. Andersson, "Microstructural Reinforcement Obtained by Diffusion Bonding", Advances in Powder Metallurgy & Particulate Materials – 2005, MPIF, Montreal, pp (2005)
7. S. Nigarura, R. Parameswaran, J. Trasorras, "Bending Fatigue of Surface Densified Gears: Effect of Root Densification Depth and Tooth Loading Mode on Fatigue Life", Advances in Powder Metallurgy & Particulate Materials 2006, San Diego, pp (2006)

8. F. Bernier, P. Boilard, J.P. Bailon, G. L'Esperance, "Machinability and Dynamic Properties of Sinter-Hardened Steel Parts, Advances in Powder Metallurgy & Particulate Materials – 2005, MPIF, Montreal, pp 201 – 210 (2005)
9. G. Piotrowski, X. Deng, N. Chawla, K. Narasimhan, M. Marucci, "Fatigue Crack Growth of Fe-0.85MO-2Ni-0.6C Steels with a Heterogeneous Microstructure", Advances in Powder Metallurgy & Particulate Materials – 2004, MPIF pp. 1-12, (2004)
10. X. Deng, G. Piotrowski, N. Chawla, "Fatigue Crack Growth of Prealloy Fe-0.85Mo-2Ni-0.6C Steels with a Homogeneous Microstructure", Advances in Powder Metallurgy & Particulate Materials – 2005, Montreal pp. 111-124 (2005)
11. MPIF Standard 56, *Standard Test Methods for Metal Powders and Powder Metallurgy Products*, Metal Powder Industries Federation, 2006 Edition, pp 101-104
12. H. Nakazawa, S. Kodama, "Statistical S-N Testing Method with 14 Specimens: JSME Method for Determination of S-N Curves", Statistical Research on Fatigue and Fracture, Current Japanese Materials Research, Vol. 2, T. Tanaka, S. Nishijima, M. Ichikawa, eds. Elsevier Science, London, pp 59-69, (1987)
13. G. Fillari, M. Marucci, "Effect of Heat Treatment and Case Carburizing High-Density P/M Steels", Advances in Powder Metallurgy & Particulate Materials – 2005, pp. 32-42, Montreal, (2005)
14. G. Fillari, T. Murphy, I. Gabrielov, "Effect of Case Carburizing on Mechanical Properties and Fatigue Endurance Limits of P/M Steels", Advances in Powder Metallurgy & Particulate Materials – 2005, Montreal, pp. 43-61 (2005)
15. H. Sanderow, oral communication (2007)
16. MPIF Standard 35 2006 Edition