ABSTRACT

Powder metallurgy processing of steel alloys typically results in a material with heterogeneous microstructure and residual porosity. The fatigue crack growth behavior of these materials is strongly affected by the nature of porosity and heterogeneous microstructure. Notched fatigue specimens were prepared from a Fe-0.85Mo prealloy mixed and binder-treated with 2%Ni and 0.6%C. The alloys were tested at three different densities: 6.98 g/cm³, 7.36 g/cm³, and 7.53 g/cm³. The microstructure at each density was characterized to determine the porosity, microconstituents, and phase fractions. Fatigue testing was performed at various R-ratios, ranging from –2 to 0.8. Increasing porosity and increasing R-ratio resulted in a decrease in $\Delta K_{th}$. In situ observation of crack growth showed that the cracks propagated through Ni-rich regions. It appears that pearlite regions, and, to some extent bainite regions, however, contributed to toughening and crack deflection.

INTRODUCTION

Steel products made by press and sinter powder metallurgy (P/M) processing are replacing products made by more conventional procedures, such as casting and forging, due to the cost savings associated with near-net shape processing [1-4]. Unfortunately, P/M parts usually have some degree of residual porosity after sintering, which can adversely affect the mechanical properties of these materials [4-8]. The nature of porosity is determined by several processing variables, including the type and amount of alloying additions, powder size distribution, green density, sintering temperature, and sintering time [1,5].

In general, an increase in porosity is associated with more irregular pores, and a greater fraction of interconnected pores. Increased pore interconnectivity and pore clustering leads to increased strain localization and damage, thus reducing the strength and ductility of the steel [5,9]. Porosity has also been shown to significantly influence the fatigue response of sintered P/M steels [4-8,10-13]. Danniger et al. [5] found that pores and pore clusters act as sites of crack initiation, while Polasik et al. [8] have shown...
that small cracks that nucleate at pores coalesce to form large fatigue cracks leading to fracture. A comprehensive understanding of the effects of porosity and microstructure on the fatigue crack growth behavior of sintered steels is required. In this study, we have examined the effect of porosity and microstructure on fatigue crack growth behavior of a Fe-0.85Mo-2Ni-0.6C at three different sintered densities: 6.98 g/cm³, 7.36 g/cm³, and 7.53 g/cm³. Quantitative characterization of the heterogeneous P/M microstructure was performed to determine the degree of porosity and nature of microstructure at all densities. With increasing density the degree of pore interconnectivity decreased, although density did not significantly affect the fraction of the microconstituents in the microstructure. Fatigue crack growth experiments were performed at constant R-ratio, ranging from -2 to 0.8. It will be shown that increasing porosity and R-ratio decrease the fatigue crack growth resistance of the material, and that the heterogeneous nature of the microstructure also influences the fatigue crack growth behavior.

MATERIALS AND EXPERIMENTAL PROCEDURE

An Fe-0.85Mo prealloy powder was blended and binder treated with 2 wt.% Ni and 0.6 wt.% graphite [14,15]. All powders were pressed into rectangular blanks and sintered at 1120ºC for 30 minutes in a 90% N₂–10% H₂ atmosphere. The samples were pressed and sintered to obtain three different sintered densities: 6.98 g/cm³, 7.36 g/cm³, and 7.53 g/cm³. The samples with 7.53 g/cm³ density were made by a double-press/double-sintering process. Porosity was determined by image analysis of several representative micrographs using both optical and scanning electron microscopy (SEM). The micrographs were segmented into black and white images, and the porosity determined by an automated procedure. Quantitative characterization of the phase fractions of the microstructure was performed by both image analysis of the optical and SEM images and Vickers hardness tests. The phase area fractions of each density were determined using a segmentation procedure with various colors, whereby each phase was assigned a specific color. The segmented images were then analyzed based on color to determine the phase fractions. Hardness measurements were conducted using a Vickers hardness indenter with a 50 g applied load. A minimum of 20 measurements were conducted in each phase.

Fatigue tests were performed on a servo-hydraulic load frame equipped with a Questar (Questar Corp., New Hope, PA) traveling microscope. The traveling microscope was used for in situ measurement and observation of fatigue crack growth. This was particularly important because it allowed visualization of the interactions between the crack and the microconstituents in the microstructure. All fatigue tests were performed using a single edge notch axial fatigue configuration [16-18]. The samples were machined by electrodisharge machining (EDM) to the following dimensions: height of 45 mm, width of 11.5 mm, thickness of 7.48 mm. The edge notch was machined by EDM to a length of 4.5 mm. Fatigue tests were performed at constant R-ratio, ranging from 0.8 to -2, in load control at a frequency of 30 Hz. A decreasing ΔK procedure was used until ΔKth was reached. At this point, the ΔK was raised to a value in the Paris law regime, and the ΔK increased to obtain the higher ΔK behavior. Samples were pre-cracked to a length of about 600 µm. The typical growth increment for a given ΔK was 200 µm, such that the increment encompassed several phases in the heterogeneous microstructure and was sufficiently larger than the plastic zone from the previous ΔK increment.

RESULTS AND DISCUSSION

Microstructure Characterization

The porosity at each density was determined from several optical micrographs using image analysis, Table 1. At the lowest density, 6.98 g/cm³, the measured porosity was 9.5%, while at the highest density 7.53 g/cm³, the porosity was about 3.9%. The values obtained from the image analysis technique
correlated well with those obtained from calculation of the sintered density from the pore-free density. The microstructure at the three densities showed noticeable differences in porosity, Figure 1. At the highest porosity, larger, more irregular, and interconnected pores were observed. With decreasing porosity, the overall pore size was smaller, and the pore shape was more regular.

<table>
<thead>
<tr>
<th>Sintered Density (g/cm³)</th>
<th>Porosity from Sintered Density (%)</th>
<th>Porosity from Image Analysis (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.98</td>
<td>10.3</td>
<td>9.5 ± 0.8</td>
</tr>
<tr>
<td>7.34</td>
<td>4.5</td>
<td>4.5 ± 0.6</td>
</tr>
<tr>
<td>7.53</td>
<td>3.2</td>
<td>3.9 ± 0.2</td>
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Figure 1. Microstructure of Fe-0.85Mo-2Ni-0.6C steels at: (a) 6.98 g/cm³, (b) 7.34 g/cm³, and (c) 7.53 g/cm³. At higher porosity the pores are larger, more irregular, and more interconnected.

Etched microstructures showed the heterogeneous nature of the microconstituents, in addition to the porosity, Fig. 2. These included coarse pearlite, fine pearlite, Ni-rich regions (likely Ni-rich austenite, surrounding the pores), and bainite, which appears at the periphery at the Ni-rich regions. Ni-rich regions likely formed as a result of incomplete diffusion of the elemental Ni into the surrounding matrix upon sintering.

Figure 2. Characteristic heterogeneous microstructure of p/m steel consisting of porosity, coarse and fine pearlite, bainite, and Ni-rich phase (likely austenite).

Quantitative characterization of the phases and phase fractions in the microstructures, at all three densities, was also conducted, Fig. 3. The phase fractions were determined from both optical and SEM micrographs. Each phase was segmented in the micrograph and indexed by a specific color. A similar analysis has been conducted by Komenda et al. [19] to characterize a heterogeneous steel microstructure. The analysis showed that the phase fractions in the microstructure were relatively uniform at all densities. As expected, the microstructure consisted primarily of coarse pearlite, with smaller amounts of fine pearlite and Ni-rich phase, and minor amounts of bainite. The bainite microconstituent was observed primarily at the periphery of the Ni-rich regions. It is interesting to note that the results from optical micrographs overestimated the amount of coarse pearlite and Ni-rich regions, at the expense of the amount of fine pearlite. This is understandable, since the fine pearlite is more difficult to resolve in the optical microscope. Thus, the true phase fraction measurements were obtained from the higher magnification SEM images.
Vickers hardness of the several microconstituents showed that the bainite regions were the hardest, followed by fine pearlite, coarse pearlite, and Ni-rich areas, Fig. 4. The relatively low hardness of the Ni-rich phase suggests that it may be retained austenite [20]. The hardness of each phase, as expected, did not vary significantly with density.

**Fatigue Behavior**

Fatigue crack growth experiments at the three densities showed that porosity had a strong effect on the crack growth rate (da/dN) and the threshold stress intensity factor, ∆K_{th}. Figure 5 shows the da/dN versus ∆K behavior for all densities, at various R-ratios. ∆K_{th} at the three densities varied between 14.7-16.4 MPa.m^{1/2} at R=-2, to 2.9-4.3 MPa.m^{1/2} at R = 0.8. These values are in the range of those found by others in similar Fe-Mo sintered steels [4,21-23]. The slope in the steady state or Paris law regime, m, was also measured [24]. Increasing R-ratio resulted in an increase in m from about 2 at low R-ratio to around 10 at R-ratio of 0.8. This increase in slope is indicative of a much higher crack growth rate, for a given ∆K, due to increasing K_{min}. We now compare the fatigue crack growth behavior, at all three porosity levels for three R-ratios: -1, 0.1, and 0.8, Fig. 6. At all R-ratios, decreasing porosity resulted in a higher ∆K_{th}. This is particularly clear at the highest R-ratio of 0.8.

The fatigue crack growth data, at all R-ratios, was analyzed using the two-parameter approach proposed by Vasudevan and Sadananda [26,27]. In the two-parameter approach there exist two critical parameters that dictate when fatigue crack propagation will take place, at a given crack growth rate, Fig. 7. The two critical components are the cyclic component ∆K and the static component K_{max}. The critical values for crack propagation then, shown in Fig. 7, are the asymptotic values in the curve, ∆K^{*} and K_{max}^{*}. By measuring the values ∆K^{*} and K_{max}^{*} over a range of da/dn values, “fatigue trajectory” plots can be generated. These provide a means to elucidate changes in material behavior with increasing crack growth rate. The two-parameter approach has also been used to categorize material behavior into different classes, Fig. 8 [26]. Materials which are dominated by environmental effects exhibit the behavior described by Class IV. Materials which are dominated by the static component, K_{max}^{*}, will show a significant monotonic component to fatigue damage, similar to that shown by the Class II material. The so-called “ideal material,” described as Class III behavior in Fig. 8, exhibits a perfect “L-shaped” behavior.
Figure 6. Effect R-ratio on fatigue crack growth, as a function of density. Increasing porosity and R-ratio resulted in a decrease in fatigue crack growth resistance.

Figure 5 da/dn versus ∆K for: (a) 6.98 g/cm³ (b) 7.34 g/cm³, and (c) 7.53 g/cm³. Porosity has a strong influence on the fatigue behavior of the P/M steel. Increased porosity and R-ratio decrease the ∆K_{TH} values of the steel. The slope of the steady-state region of curves was also found to increase with R-ratio, indicative of an increased monotonic contribution to fatigue.

Figure 7. Schematic of two parameter approach to fatigue (after Vasudevan and Sadananda [27]). Two critical parameters are required for crack growth, a cyclic component, ∆K, and a static component, K_{max}.
We begin by examining the $\Delta K$ versus R-ratio behavior. The curves are relatively linear, although it can be seen that with increasing density, a higher $\Delta K$ is required to achieve a given crack growth rate. Also, at least up to an R-ratio of 0.8, there does not seem to be a plateau in $\Delta K$, as described by the Class III behavior. Rather, it is likely that large degree of plasticity is present at the crack tip at very high R-ratio, that makes the P/M materials fall into the Class II category. The changes in material behavior with increasing R-ratio and with increasing crack growth rate are more easily observed in a series of plots of $\Delta K$ versus $K_{\text{max}}$, Fig. 9. A series of curves are shown at each of the crack growth rates, shown in Fig. 10. As the degree of porosity increases, the monotonic contribution to the fatigue crack growth behavior increases. This can be seen graphically as the deviation from the horizontal asymptote in the $\Delta K$-$K_{\text{max}}$ plots. The behavior can be rationalized by the fact that with increasing R-ratio, the localization of strain and plasticity at the crack tip are enhanced in a material with a higher degree of porosity. Thus, a lower threshold is required for crack growth to take place with a larger degree of porosity at the crack tip. This behavior is exacerbated with increasing crack growth rate, as shown by the increased deviation of the “L-shaped” curves with increasing crack growth rate. This behavior is more predominant at the lower densities, because of the enhanced effect of porosity. At the highest density, the material behavior is closer to the classical “ideal material.” To quantify the degree of monotonic superposition, a comparison between the experimental data and a theoretical pure fatigue line was made. The condition for “pure fatigue” is that $\Delta K^* \text{ equal } K_{\text{max}}^*$, Fig. 11(a). Thus, any deviation from this 45° line, must be due to environmental, crack closure, or static effects. As the line moves toward the horizontal axis, this signifies an increased contribution from monotonic loading, since $K_{\text{max}}$ is the dominating component to fatigue [25]. Our data show that at the two lower densities the behavior is close to parallel to the horizontal axis, indicating significant static effects associated with strain localization between pores, Fig. 11(b). At the highest density, however, the line is close to parallel to the pure fatigue line. This indicates that the static contribution with increasing R-ratio is not as predominant. Rather, the offset between the two parallel lines may be attributed to two components: (a) fatigue crack closure at low R-ratio and (b) static effect at higher R-ratio.

![Figure 8. Classification of different types of materials by the two parameter approach. Class I materials exhibit limited sensitivity to environmental effects. Class III material is termed the “ideal material” behavior, while Class II materials are influence by large plasticity at the crack tip.](image)

![Figure 9. $\Delta K$ versus R for: (a) 6.98 g/cm³, (b) 7.34 g/cm³, and (c) 7.53 g/cm³.](image)
Figure 10. Trajectory maps of $\Delta K$ versus $K_{\text{max}}$ at various $\text{da/dN}$: (a) 6.98 g/cm$^3$, (b) 7.34 g/cm$^3$, and (c) 7.53 g/cm$^3$. Note that for highest porosity, the horizontal asymptote is diminished due to the significant contribution of plasticity at the crack tip (from $K_{\text{max}}$).

Figure 11. Quantifying the Degree of Monotonic Superposition: (a) monotonic contribution quantified by measuring the deviation of the experimental data from the “pure fatigue” line, and (b) as porosity increases, the degree of monotonic superposition also increases. This is indicated by the increasing deviation from the pure fatigue line.
In this section we describe fractographic analysis after fatigue crack growth. Since the crack path appeared quite tortuous, due to the heterogeneous microstructure of these steels, we have also quantified the degree of crack deflection on the measured $\Delta K$, per the model of Suresh [28]. The crack appears to be highly dependent on the phase(s) at the crack tip, Fig. 12. For the Ni-rich regions, cracks tend to propagate in a linear fashion, suggesting that the Ni-rich regions offer little to no resistance to crack propagation. This is further by the Vickers hardness data, which showed the Ni-rich phase to be very soft, indicating that it might be Ni-rich austenite phases [20]. For the pearlite regions, cracks tend to be highly deflected, with some evidence of the Fe$_3$C particles in the ferrite matrix bridging the crack, Fig. 13. Pores inside the Ni-rich regions can also act as nucleation sites for secondary cracks. It has been demonstrated that these cracks originating at pores ahead of the crack tip, often join the main crack [8,13], Fig. 14.

Cracks propagating through the coarse pearlite, fine pearlite, and bainite all show large increases in the degree of fatigue resistance due to crack deflection, Fig. 14. Crack arrest is often present, and further induces crack deflection through branching. The role of the Ni-rich areas in fatigue is still not quite clear. Quantitative measurements of the crack growth rate in the different microstructural constituents are being conducted, and will shed some light on the role of Ni-rich areas.

Figure 12. Fatigue Crack Behavior through heterogeneous microstructure: (a) Ni rich, and (b) Pearlite. The crack propagates through the Ni-rich areas, but it tortuous and deflected through pearlite due to the Fe$_3$C needles.

Figure 13. Crack Bridging due to Fe$_3$C: (a) Fe$_3$C needles pulled out of the ferrite matrix during fatigue, and (b) EDS analysis showing the composition corresponding to Fe$_3$C.
The crack deflections induced by the heterogeneous microstructure increased the fatigue resistance of the steel. The increase in the fatigue resistance of the steels can be quantified by applying the crack deflection model by Suresh [28]. The model assumes that for a given crack length there exist linear portions, S, and deflected portions, D, Fig. 15. The angle between the linear portion and the deflected portion is given by $\theta$. By measuring S, D and $\theta$, the true $\Delta K$, corrected for crack deflection, can be calculated from the following equation:

$$
\Delta K_{I,app} \approx \Delta K_{eff} \left[ \frac{D \cos^2 \left( \frac{\theta}{2} \right) + S}{D + S} \right]^{-1} \tag{1}
$$

The values of $\Delta K_{I,app}$ calculated from the use of the Suresh deflection model represent increases in fatigue resistance due to crack deflection. The corrected $\Delta K$ values are shown in Fig. 16. Note that the curves shift to the right, indicating some contribution from deflection, although this contribution is quite small. Also, the contribution from deflection does not appear to be noticeably different between the three densities. This may be due to the fact that all three densities exhibit relatively equivalent heterogeneous microstructures.

Figure 14. In situ observation of cracking during fatigue: (a) Initial, (b) 10,000 cycles, (c) 16,000 cycles, and (d) 22,000 cycles.

Figure 15. Suresh’s model [28] for quantifying crack deflections. The crack consists of linear regions, S, and deflected regions, D. The angle between S and D is $\theta$. 

$$
\Delta K_{I,app} \approx \Delta K_{eff} \left[ \frac{D \cos^2 \left( \frac{\theta}{2} \right) + S}{D + S} \right]^{-1} \tag{1}
$$
CONCLUSIONS

In this study the effect of porosity and heterogeneous microstructure on the fatigue crack growth behavior of sintered steels was systematically studied. The following conclusions can be made based on our results:

- The heterogeneous microstructure plays an important role in fatigue crack behavior, in particular, porosity significantly influences fatigue crack growth.
- Increasing porosity and R-ratio resulted in a decrease in $\Delta K_{TH}$ values. Increased R-ratio and porosity also increased the monotonic contribution during fatigue due to strain localization between pores at the crack tip.
- The fatigue crack path is tortuous and highly dependent on microstructure. Areas of pearlite cause crack arrest, deflection and branching, while Ni-rich and porous areas do not appear to offer resistance to crack growth.
- Crack deflections increase the fatigue resistance of the material for all three densities, although the contribution from crack deflection is quite small.

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REFERENCES