FATIGUE CRACK INITIATION AND PROPAGATION IN FERROUS POWDER METALLURGY ALLOYS

S.J. Polasik, J.J. Williams, and N. Chawla

Department of Chemical and Materials Engineering
Arizona State University
Tempe, AZ 85287-6006

K.S. Narasimhan

Hoeganaes Corp.
1001 Taylors Lane
Cinnaminson, NJ 08077

ABSTRACT

Many of the targeted applications for powder metallurgy materials, particularly in the automotive industry, undergo cyclic loading. It is, therefore, essential to examine the fatigue mechanisms in these materials. The mechanisms of fatigue crack initiation and propagation in ferrous powder metallurgy components have been investigated. The fatigue mechanisms are controlled primarily by the inherent porosity present in these materials. Since most, if not all, fatigue cracks initiate and propagate at the specimen surface, surface replication was used to determine the role of surface porosity in relation to fatigue behavior. Surface replication provides detailed information on both initiation sites, and propagation path of fatigue cracks. The effect of microstructural features such as pore size, mean pore spacing, as well as the heterogeneous microstructure on crack deflection was examined and is discussed. Fracture surfaces were examined to elucidate a mechanistic understanding of fatigue processes in these materials.

INTRODUCTION

P/M components are increasingly replacing wrought materials in high performance applications. Many of the targeted P/M applications, particularly in the automotive industry, involve cyclic loading. Thus, an understanding of fatigue mechanisms in these materials is essential. IN diffusion alloyed powder bonding between iron and the alloying particles is promoted during an annealing step, typically at an intermediate temperature for partial diffusion to take place, prior to pressing and sintering. Binder treatment of the powder mixture prior to pressing and sintering is a new and effective technique to minimize segregation and eliminates the diffusion-alloying step [1-4]. Advances in binder treated versus conventional diffusion alloyed powders have been driven by the cost effectiveness of binder treatment, particularly since the annealing step is eliminated. In the binder treated process, a polymeric binder mechanically bonds the alloying additions to the larger iron particles, resulting in a smaller path for diffusion during sintering and enhanced densification upon sintering. In order to avoid large-scale residual porosity or harmful by-products, the binder is completely burned out prior to sintering. Burnout is accomplished either by a debinding step (at an intermediate temperature below the sintering temperature), or by heating at a relatively slow rate until the sintering temperature is reached.

There are many other advantages of binder-treatment over conventional P/M processes. For example, faster and more consistent flow into the die cavity and increased green strength from
better bonding of particles takes place by binder treatment. Additionally, there is a reduction of fine particle dusting, resulting in more efficient use of alloy powders and plant cleanliness. Furthermore, a pre-alloyed powder, with less compressibility than elemental powders can be used since the binder improves lubricity, and therefore compressibility of the powder [4].

The microstructure of the binder treated or diffusion alloyed materials is characterized by a porous and heterogeneous microstructure that develops from incomplete diffusion of alloying elements during sintering. Due to this incomplete diffusion of alloying elements, a variety of phases are formed. Several investigators have shown that porosity adversely affects the mechanical properties of P/M components [5-13]. Under monotonic tensile loading, porosity reduces the effective load bearing cross-sectional area [7], decreasing both strength and ductility [10]. Two types of porosity are typically observed in P/M materials: interconnected and isolated porosity. Interconnected porosity has a more pronounced effect on ductility than isolated porosity. Isolated pores imply a larger contribution from the original powder particles and thus a larger deformed volume, effectively increasing the macroscopic ductility. With interconnected porosity, on the other hand, there are small sinter bonds between particles that fail by highly localized ductile rupture, thus reducing macroscopic ductility of P/M materials [6,10]. In this manner, much of the metallic phase does not contribute to mechanical strength. A useful analogy describing the influence of interconnected porosity on mechanical strength is proposed by Danninger et al. [6]. They compare a P/M steel with interconnected pores with a chain composed of intermittent thick (heavy) and thin (light) links. The weight of the chain may be determined by the heavy links, but the strength is limited by the thin links.

Porosity significantly affects fatigue performance, although the role of porosity in fatigue is somewhat different than that in tension. In many investigations [5,6,8,10,14-16], the preferred site for crack initiation occurred at pores or pore clusters located at or near the surface or the specimen. Holmes and Queeney [7] proposed that the relatively high stress concentration at pores, particularly surface pores, is responsible for localized slip leading to crack initiation. In general, an angular pore creates a higher stress concentration and stress intensity factor than a round pore [10]. Christian and German [9] showed that total porosity, pore size, pore shape, and pore separation are important factors that control the fatigue behavior of P/M materials. Pores have also been proposed to act as linkage sites for crack propagation through interpore ligaments [8]. Recent limited data indicate that binder-treatment has equivalent tensile and fatigue properties to those of diffusion alloyed materials [1,4].

Since the preferred initiation site for fatigue cracks is frequently the specimen surface, surface replication is an ideal means to study crack initiation and growth during fatigue [9,17-19]. Thus, we have used surface replication as a means to studying the role of surface porosity during fatigue of P/M materials. Surface replication allows the determination of crack initiation and the nature and duration of crack propagation during the fatigue process.

Surface replication is a particularly valuable tool to investigate short fatigue cracks. Short fatigue cracks are on the order of microstructural features such as grains or inclusions. The study of short cracks is very important because they often propagate at much faster rates than long fatigue cracks (typically greater than 2mm) under the same driving force. Additionally, short cracks propagate at stress intensities below the long-crack stress-intensity factor, ΔKth, determined using a linear elastic fracture mechanics (LEFM) approach. Since cracks that grow until they reach the critical size needed for failure must have gone through a period when they can be considered short cracks, long crack growth data alone is therefore not sufficient to describe fatigue behavior.
or predict fatigue life [15, 20]. In this paper we have conducted a detailed examination of fatigue crack initiation and propagation mechanisms of Fe-0.85Mo prealloy, binder-treated P/M materials. By using surface replication and fractographic techniques, a detailed understanding of fatigue processes in these materials was obtained.

MATERIALS AND EXPERIMENTAL PROCEDURE

Alloys of Fe-0.85Mo-1.5Cu-1.75Ni-0.6 graphite were fabricated by binder treating a mixture of pre-alloyed Fe-0.85Mo with the alloying additions. Binder treatment was conducted using a proprietary process developed by Hoeganaes Corp. [2,3]. All powders were pressed into rectangular blanks, to a green density of 7.0 g/cm$^3$. Sintering was conducted at 1120ºC for 30 minutes in a 90% N$_2$–10% H$_2$ atmosphere. The as-sintered microstructure of the P/M alloys was examined by etching with a 2% Nital solution. Digital image analysis techniques were used to determine the pore morphology (pore size and shape distribution as well as mean pore spacing) of the material. Tensile tests were conducted in strain control at a constant strain rate of $10^{-3}$/s. Cylindrical axial fatigue specimens were machined from the sintered rectangular blanks. Fatigue testing was conducted on a servohydraulic load frame in load control, an R ratio ($\sigma_{\text{min}}/\sigma_{\text{max}}$) of –1, and a frequency of 40 Hz (40 cycles per second). The cylindrical axial fatigue geometry, dimensions, and testing procedures are given elsewhere [21].

Fatigue specimens were hand polished, using diamond paste, to a 1µm finish. Surface replication was conducted by interrupting the fatigue test intermittently placing the sample under a small tensile load (~ 0.25$\sigma_{\text{max}}$) to avoid closure of any fatigue cracks during the replicating procedure. The entire gauge section was then bathed with acetone and subsequently covered with cellulose acetate tape. After the solvent (acetone) evaporated and the replicating tape dried, the replicas were flattened onto double-stick tape and placed between two microscope slides. Using an optical microscope, digital micrographs were taken of the entire replicated surface for the various fatigue cycles. The largest crack was identified on the last replica prior to failure, and this crack (or cluster of cracks) was identified on prior replicas until the point of crack initiation. With the current surface replication technique, a crack of length greater than 15µm could be identified.

RESULTS AND DISCUSSION

Microstructure

During sintering a variety of phases are formed due to localized variations in composition. The microstructure in the as-sintered condition of the Fe-0.85Mo-1.5Cu-1.75Ni-0.6 graphite alloy is shown in Figure 1. Without etching, only porosity is observed (Figure 1a). Upon etching, a heterogeneous microstructure consisting of martensite, nickel-rich ferrite, divorced pearlite (“divorced” since it is not lamellar, as in conventional pearlite), and nickel-rich regions surrounding pores is observed.

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Figure 1. Micrographs of P/M material tested in as-sintered condition: (a) unetched and (b) etched. Note the primary and secondary porosity as well as the heterogeneous nature of the microstructure.

The porosity is a combination of primary porosity (always present after sintering these materials) and “secondary porosity.” Secondary porosity consists of residual porosity from liquid phase formation and diffusion of alloying additions, such as copper, at the sintering temperature. In order to characterize the pore structure, both the pore size and shape distribution were determined from optical micrographs of cross-sections of the samples. Pore size was estimated by measuring the pore area. Pore shape was then characterized using a shape form factor, $F$:

$$F = \frac{4\pi A}{P^2}$$

where $A$ is the measured pore area and $P$ is the measured pore perimeter. For the shape form factor, a value of “1” denotes a perfectly round pore, and values that approach zero denote increasingly irregular pores. Results of the pore size and shape analyses are shown in Figure 2.
The pore size and shape analyses show that the P/M material tested consisted mainly of pores below 300 μm$^2$ in area and relatively irregular shape (most shape factors are between 0.4 and 0.7). The irregularity of the pores suggests a greater amount of local stress concentration and subsequently, a larger amount of potential fatigue crack initiation sites than in a P/M material with perfectly spherical porosity.

**Tensile Behavior**

The tensile stress-strain behavior of the Fe-0.85Mo-1.5Cu-1.75Ni-0.6 graphite alloy is shown in Figure 3. Table 1 summarizes the tensile properties of this alloy. Porosity clearly decreases the macroscopic ductility due to localization of ductile rupture in the sinter bonds. The hard regions shown in Figure 1 could contribute to a higher work hardening rate and lower ductility than in wrought materials.

![Tensile stress-strain behavior](image)

**Figure 3.** Tensile stress-strain behavior of the binder-treated alloy.

<table>
<thead>
<tr>
<th>Elastic Modulus (GPa)</th>
<th>0.2% Offset Yield Strength (MPa)</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>Strain-to-failure (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>121</td>
<td>526</td>
<td>774</td>
<td>1.83</td>
</tr>
</tbody>
</table>

*Table 1. Tensile Properties of Fe-0.85Mo binder-treated alloy*
As is true with most P/M materials, a combination of localized ductile and brittle fracture was observed, Figure 4. Ductile rupture, due to microvoid coalescence occurred locally in isolated sinter bonds and brittle failure generally occurred in large pearlitic grains. Damage was more predominant in the form of localized ductile rupture of sintered necks, since these necks see mto have lower strength than larger, brittle pearlitic grains.

![Figure 4. SEM micrographs of tensile fracture: (a) ductile rupture in localized sinter bonds and (b) cleavage in large pearlitic grains.](image)

**Fatigue Behavior**  
The stress versus cycles behavior of the Fe-0.85Mo-1.5Cu-1.75Ni-0.6 graphite P/M steel is shown in Figure 5 (fatigue run-out was taken as $10^7$ cycles). The results of the present investigation are compared to a similar alloy with 0.5% Mo, studied by Chawla et al. [1,4]. Notice the clear increase in fatigue resistance with an increase in Mo content. An increase in solid solution strengthening by increasing barriers for dislocation motion and cyclic slip likely account for this behavior.

![Figure 5. Comparison of binder treated alloys with 0.5% Mo and 0.85% Mo. An increase in Mo results in an increase in fatigue life.](image)
The stress-strain behavior was also quantified during fatigue. The width of the stress-strain hysteresis loop was used to determine the cyclic plastic strain amplitude and the slope of the loop, i.e., the secant modulus, was used to estimate fatigue damage. In this analysis a useful parameter to quantify the evolution of damage is the damage parameter [22], \( D_E \), defined as

\[
D_E = 1 - \frac{E}{E_o}
\]

where \( E \) is the secant modulus at any given number of cycles and \( E_o \) is the elastic modulus of the as-sintered material. Changes in the plastic strain amplitude and damage parameter can be used to shed some light into the processes of cyclic hardening or cyclic softening operating during fatigue. In the low cycle fatigue (LCF) regime, the plastic strain amplitude showed a slight decrease very early in the fatigue life, indicating cyclic hardening, followed by a gradual increase, i.e., cyclic softening, until failure took place, Figure 6. The damage parameter, however, does not seem to be as sensitive to cyclic hardening and softening as the plastic strain.

![Damage Parameter and Plastic Strain](image)

Figure 6. (a) Damage parameter and (b) plastic strain data from stress-strain hysteresis measurements during fatigue. In both cases, (a) and (b), the LCF regime exhibits cyclic hardening and gradual cyclic softening while in the HCF regime remains relatively unchanged. \( N_i \), the number of cycles to crack initiation, for the LCF specimen is indicated.

In the high cycle fatigue regime (HCF), both the damage parameter and plastic strain amplitude remained relatively unchanged since the local stresses and strains are more elastic than in the LCF regime. For the LCF specimen, the cycle at which crack initiation occurred in a similar sample at the same stress level was determined from surface replication and is indicated in the
figure. It is interesting to note that the onset of crack initiation correlates fairly well with the point at which $D_E$ shows a noticeable increase.

As mentioned above, one of the most important controlling factors in the fatigue resistance of P/M materials is porosity. Fatigue cracks tend to initiate near pores or pore clusters because of the higher localized stress intensity associated with these defects. Pores with a more irregular shape have higher local stresses and stress intensities than smoother, rounded pores. Typically, cracks initiate at pores located at or near the surface of the specimen, because the stress intensity is higher than for a pore within the interior [13]. The number of cycles required for crack initiation, $N_i$, was determined from surface replication, and the fraction of the life of the specimen spent initiating a crack, $N_i/N_f$, was plotted as a function of cycles to failure, $N_f$, Figure 7. This approach provides a quantitative estimate of the fraction of fatigue life spent in initiation and propagation. There are two pronounced differences between the LCF and HCF regimes; cracks initiate very early (15%) in LCF, yet initiation life accounts for more than 80% in HCF.

![Figure 7](image)

Figure 7. Ratio of cycles to initiation to cycles to failure. Note that in LCF regime a significant fraction of life is spent in crack propagation, while in the HCF regime, most of the fatigue life is spent in crack initiation.

After a crack has initiated at surface or subsurface pores, it tends to propagate and grow through the interpore ligaments, using pores as linkage sites, as seen in Figure 8. While other investigators have proposed microcrack linkage as a mechanism, from fractographic observations [15,23], surface replication allows *in situ* monitoring of the crack initiation and growth process. The initiation and linkage of short fatigue cracks is monitored during cyclic loading, as opposed to post-mortem fractography. The surface replication technique used in this study confirms the linkage of smaller cracks to form one final crack that causes failure of the material.
Figure 8. Crack propagation illustrating the role of pores as crack linkage sites (arrows indicate loading axis).
Surface replication provides invaluable information on short fatigue cracks, which behave differently than long fatigue cracks. As an example, the stress intensity ranges and corresponding crack growth rate per cycle were calculated. Crack measurements from surface replication were used to determine crack growth rates of the small cracks. The stress intensity, $K$, was calculated using the expression for a surface crack in a cylinder (seen in Figure 9) developed by Newman and Raju [24],

$$\Delta K = F\Delta \sigma \sqrt{\pi \frac{a}{Q}} \tag{3}$$

where $a$ is the crack depth, $Q$ is the shape factor for an ellipse (defined as $1 + 1.464(a/c)^{1.65}$ for $a/c \leq 1$), $\Delta \sigma$ is the tensile component of the fatigue stress, and $F$ is a boundary correction size factor that accounts for crack size, crack shape, and the ratio of crack size to specimen diameter. Note that only the tensile component of the fatigue stress range is used to compute $\Delta K$ since only tensile loads result in crack growth. Newman and Raju calculated the values for $F$ using finite element modeling (FEM). A third order polynomial was fitted to the $F$ values given in [24] and these projected values were used to calculate the stress intensity at the interior of the crack tip, assuming the crack had an $a/c$ of 0.6.

![Figure 9. Schematic of surface crack in cylindrical rod used for calculations of stress intensity of fatigue cracks.](image)

The value for the $a/c$ ratio used for the calculations in this study was determined from an SEM micrograph of a fracture surface, Fig 10. The initiation site was identified and the angle, $\theta$, between two radii of a circle superimposed on the fracture surface was measured. The arc length, $2c$, was calculated using the following equation

$$2c = \left(\frac{\theta}{360}\right)\pi D \tag{4}$$

where $D$ is the diameter of the specimen. Finally, $a$ was measured and $a/c$ was calculated to be 0.58, or about 0.6.

Figure 11 illustrates crack growth in LCF and HCF regimes. Due to crack deflection, the value for the crack length ($2c$) was taken as the horizontal projection of the crack (the length of the crack was taken to be perpendicular to the loading axis). The $da/dN$ data in Figure 11 shows that while several cracks initiate and link together, some fatigue cracks show a deceleration in crack growth rate, followed by arrest. Conversely, some cracks decelerate, but grow subsequently. Crack arrest or deceleration may be attributed to microstructural barriers such as grain boundaries, locally hard regions (such as Ni-rich regions), interaction with other cracks, or even porosity. In addition, many of these cracks measured through surface replication are small.
cracks and the plastic zone is of comparable size to the crack, requiring high stresses for propagation [25]. Crack deflection may also take place due to microstructural barriers in the material.

Figure 10. SEM fractograph used to determine a/c crack ratio used for stress intensity calculations.

Figure 11. da/dN versus stress intensity ranges (ΔK) as calculated using Raju and Newman analysis [24] for (a) LCF and (b) HCF.
Surface replication also enabled the identification of the initiation area on the fracture surface, through scanning electron microscopy (SEM). A SEM micrograph of a fatigue crack initiation site is shown in Figure 12. The intermittent nature of fatigue crack growth during each cycle can be observed through the existence of localized fatigue striations in various broken sinter bonds, Fig. 13. Striations occur when plastic strains at the crack tip cause localized slip on planes of maximum shear. The crack front therefore undergoes repetitive blunting and sharpening during propagation during both the tensile and compressive portion of the cycle. Unlike striations in wrought materials, the striations in P/M materials are highly localized. This is due to the multiple sites of favorable orientation and size that undergo blunting and sharpening and give the step-like appearance of the striations.

Ductile rupture is also present on the fatigue fracture surface, as seen in Figure 13. The ductile failure regions are highly localized in sinter bonds and develop from microvoid coalescence.
The localized necking and microvoid growth, while present in the crack initiation stage, are predominately found in the later stages of fatigue. Small spherical inclusions, which likely contributed to microvoid formation, were found at the bottom of some microvoids, Figure 13. The composition of the inclusions was identified as MnS, as determined by energy dispersive spectroscopy (EDS). Cleavage fracture was also observed on the fracture surface. Since this type of failure was seen in the tensile tests and therefore fast fracture, cleavage failure was associated with the fracture surface region corresponding to the final, fast crack propagation leading to failure. Generally when this cleavage fracture occurred, it was found to be in large pearlitic grains, similar to what is shown in Figure 4.

CONCLUSIONS

The following conclusions can be made concerning the fatigue behavior of binder-treated Fe-0.85Mo-1.5Cu-1.75Ni-0.6 graphite alloys:

- The microstructure of the alloy consisted of a heterogeneous microstructure with areas of “divorced” pearlite, martensite, and nickel-rich ferrite. In addition conventional porosity was accompanied by secondary porosity formed due to the copper particles forming a liquid phase and diffusing into Fe particles during sintering. The pores had relatively irregular shape.

- An increase in Mo content increases the fatigue resistance of the alloy, presumably due to increased solid solution strengthening and the hindering of cyclic slip mechanisms at pore tips.

- The material showed cyclic hardening followed by a gradual cyclic softening, being most pronounced in LCF. Additionally, the approximate cycles to crack initiation, determined by surface replication of a similar specimen at the same stress level, coincided with the beginning of the rapid increase in plastic strain and damage parameter in the LCF regime. Thus, a significant increase in the damage parameter can be correlated to small crack initiation.

- The fraction of the life required to initiate a fatigue crack, $N/N_f$, tends to increase with a decrease in fatigue stress amplitude. Thus, crack initiation occurs early in the life of LCF specimens and crack propagation mechanisms, such as deflection due to microstructural features, are more significant. In contrast, since crack initiation occupies a much higher fraction of fatigue life in the HCF regime, porosity has a more predominant effect in this regime.

- Short fatigue cracks initiate at surface pores or pore clusters and propagate at faster rates and lower stress intensities than long fatigue cracks. Linkage of these small fatigue cracks to form the final critical size crack that causes failure was documented by in situ surface replication.

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