ABSTRACT

The influence of Cu and Ni on tensile and fatigue behavior of Fe-0.85Mo prealloy with 0.6% graphite addition was investigated. The transient liquid phase formed by the presence of Cu resulted in more rounded pores in the Fe-Ni-Cu alloy compared to Fe-Ni. While the total porosity was similar in both alloys, a larger fraction of secondary pores was present in the Fe-Ni-Cu. The addition of Cu resulted in an increase in proportional limit stress, ultimate tensile strength, and fatigue strength over the Cu-free alloy. The higher fatigue resistance was attributed to the enhanced solid solution strengthening provided by Cu in Fe, and the higher proportional limit in the Fe-Ni-Cu alloy, caused by the more rounded nature of the pores, and stronger Fe-Ni-Cu matrix surrounding the pores. The proportional limit stress appears to be a good indicator of fatigue strength, since it quantifies the onset of localized plasticity in these materials.

INTRODUCTION

Sintered ferrous alloys are frequently alloyed with Cu or Ni for enhanced sinterability and mechanical properties [1-5]. The two alloying elements play very different roles during the sintering process, particularly because Cu has a much lower melting point than Ni. Thus, at the typical temperatures used to sinter ferrous alloys, Cu forms a liquid phase while Ni remains in the solid state. The implication of Cu being in liquid phase is that when the Cu particles melt, they leave their original positions, creating very small pores in their wake [6]. This type of porosity is characterized as secondary porosity, because the pores are much smaller than the larger primary pores associated with the sintering of ferrous alloys. Another effect attributed to Cu liquid phase is that it forms a semi-permeable liquid layer around the Fe particles, which improves iron particle bonding but also reduces carbon diffusion into iron [5]. Since Ni remains in the solid state during sintering, partial diffusion of Ni into Fe contributes to a heterogeneous microstructure, consisting of nickel-rich ferrite as well as nickel-rich areas predominantly at the periphery of the pores [4].

In this study we have attempted to separate the effects of Cu and Ni on the tensile and fatigue properties of Fe-0.85Mo prealloy steels. Two alloys were studied, both containing Ni, but only one containing Cu. Microstructure characterization, including pore size and pore shape characterization, was performed to better understand the tensile and fatigue behavior of the alloys. It will be shown that the combination of Cu and Ni provides better tensile and fatigue properties than Ni alone. The effect of Cu on tensile and
fatigue behavior will be discussed in terms of: (a) Cu as a strengthening agent in the Fe particles and (b) Cu liquid phase effect on pore morphology, size, and distribution.

EXPERIMENTAL PROCEDURE

Two Fe-0.85Mo alloys were investigated in this study, both containing approximately the same amount of Ni and graphite, Table 1. The Fe-Ni-Cu alloy contained 1.5% of copper, while the Fe-Ni alloy did not contain any copper.

Table 1. Composition of powder metallurgy alloys examined in this study.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Mo</th>
<th>Cu</th>
<th>Ni</th>
<th>C</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-Ni-Cu</td>
<td>0.85</td>
<td>1.50</td>
<td>1.75</td>
<td>0.60</td>
<td>Balance</td>
</tr>
<tr>
<td>Fe-Ni</td>
<td>0.85</td>
<td>0.00</td>
<td>2.00</td>
<td>0.60</td>
<td>Balance</td>
</tr>
</tbody>
</table>

The powders were blended and binder treated using a proprietary process developed by Hoeganaes Corp. [7,8]. All powders were pressed into rectangular blanks, to a green density of 7.0 g/cm³, and sintered at 1120°C for 30 minutes in a 90% N₂–10% H₂ atmosphere. In order to characterize the pore structure, both the pore size and pore shape distributions were quantified. Analysis of primary pores (taken as pores with areas above 25 µm²) was taken from digital image analysis of optical micrographs of the samples’ cross-section. The smaller, secondary pores (taken as pores with areas smaller than 25 µm²) were analyzed by scanning electron microscopy (SEM). Pore size was estimated by measuring the pore area fraction, while pore shape was characterized by a pore shape factor, $F$ [5]:

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

where $A$ and $P$ are the measured pore area and measured pore perimeter, respectively. In the analysis of the pore shape factor a value of 1 denotes a perfectly circular pore, and values that approach zero denote increasingly irregular pores.

Tensile and fatigue testing were conducted on cylindrical axial specimens machined from the sintered rectangular blanks. The specimens had an overall length of 75 mm, gage length of 20 mm, and gage diameter of 4.75 mm. Specimens were hand polished, using diamond paste, to a 1 µm finish. Tensile tests were conducted at a constant strain rate of $10^{-3}$/s, while axial fatigue testing was conducted at an R ratio ($\sigma_{\text{min}}/\sigma_{\text{max}}$) of –1, and a sinusoidal frequency of 40 Hz. All mechanical testing was conducted on a precision aligned servohydraulic load frame. SEM and energy dispersive spectroscopy (EDS) were performed for fracture surface analysis and compositional analysis, respectively.

RESULTS AND DISCUSSION

The sintered density of both alloys was about 7.0 g/cm³ and the dimensional change after sintering was also similar, Table 2. Microstructural analysis revealed that porosity in both alloys to be about 12%, which was consistent with the density of the alloy relative to the pore-free density. While the total porosity was similar, optical micrographs of both alloys, Fig. 1, revealed a larger fraction of secondary pores in the Cu-containing alloy. This was to be expected, as mentioned in the introduction, since the Cu formed a transient liquid phase during sintering and left the secondary pores in its wake.

Table 2. Sintered properties of Fe-0.85Mo alloys.
<table>
<thead>
<tr>
<th>Alloy</th>
<th>Green Density (g/cm³)</th>
<th>Sintered Density (g/cm³)</th>
<th>Dimensional Change (%)</th>
<th>Pore area fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-Ni-Cu</td>
<td>7.05</td>
<td>7.01</td>
<td>-0.06</td>
<td>12.0</td>
</tr>
<tr>
<td>Fe-Ni</td>
<td>6.97</td>
<td>6.98</td>
<td>0.02</td>
<td>12.1</td>
</tr>
</tbody>
</table>

The absence of a large amount of dimensional change in the Cu-containing alloy can be explained by the competing mechanisms of (a) transient liquid phase formation and diffusion of this phase into Fe particles [6] and (b) formation of secondary pores formed by the liquid phase [9]. The absence of liquid phase in the Fe-Ni alloy resulted in an almost negligible dimensional change. Scanning electron microscopy of the polished surfaces further indicated that Ni did not diffuse completely into iron and was present predominantly at the periphery of the primary pores in both alloys, Fig. 2.

![Figure 1](image1.png)

**Figure 1.** Optical micrographs of as-sintered microstructure: (a) Fe-Ni-Cu and (b) Fe-Ni alloy. Note the higher degree of secondary porosity in the Cu-containing alloy.

![Figure 2](image2.png)

**Figure 2.** Backscattered scanning electron micrograph showing nickel-rich areas from incomplete diffusion of Ni (bright regions in the micrograph), predominantly around primary pores.

The pore size and distribution analysis quantified primary pores (> 25 µm²) and secondary pores (< 25 µm²) separately. A comparison of the primary pore size and pore shape distribution for both alloys is...
shown in Fig. 3. The Fe-Ni-Cu alloy had a lower frequency of pores between 25-75 µm, but a larger frequency of pores above 75 µm. Thus, from the pore size distribution, the primary pores in the Fe-Ni-Cu alloy appeared to be larger. Pore shape analysis, Fig. 3(b), indicated that these pores were also slightly rounder than those in the Fe-Ni alloy. Analysis of secondary pores, which required microstructure characterization using SEM, is shown in Fig. 4. The pore size was normalized, due to the proprietary nature of the Cu particle size used in these alloys. The analysis indicated that a larger fraction of secondary pores was present in the Fe-Ni-Cu alloy, and these pores were also more rounded than those in the Fe-Ni alloy. These pores may be attributed to the voids left behind by the original Cu particles [9]. While the pore shape analysis was useful, it did not reflect the dependence of pore shape on pore size. Analysis of pore shape for the distribution of pore sizes, Fig. 5, indicated that for a given pore size the pores in the Fe-Ni-Cu alloy were more rounded than that in the Fe-Ni alloy.

Figure 3. Pore size and pore shape distribution of primary pores (larger than 25µm²), revealing larger and slightly rounder pores in Cu-containing alloy.

Figure 4. Pore size and pore shape distribution of secondary pores (smaller than 25µm²), showing higher density of secondary pores and rounder pores in Cu-containing alloy.
The tensile behavior of the alloys was profoundly influenced by the presence of Cu, Table 3. The Cu-containing alloy exhibited higher proportional limit stress (the stress defined as the onset of plasticity in the material, taken here as 1% deviation in linearity of the stress-strain curve) and higher ultimate tensile strength. The Young’s moduli of the alloys were similar, since both alloys contained the same relative porosity. The higher tensile strength in Fe-Ni-Cu can be attributed to the presence of Cu in solid solution, which is effective in increasing the resistance to dislocation motion and increasing the work-hardening rate of the alloy. The higher proportional limit was also affected by the presence of Cu in solid solution, but may have been more influenced by the more rounded nature of pores in the Fe-Ni-Cu. The stress concentration at a spherical pore is clearly less severe than that of a very irregular pore. Fractographic analysis of tensile fracture surface of both alloys was characteristic of most sintered ferrous powder metallurgy alloys, Fig. 7, showing evidence of localized ductile rupture of sintered bonds.

Table 3. Tensile behavior of Fe-Ni-Cu and Fe-Cu alloys.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Young’s Modulus (GPa)</th>
<th>Proportional Limit Stress (MPa)</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>Strain-to-Failure (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-Ni-Cu</td>
<td>135</td>
<td>221</td>
<td>797</td>
<td>1.91</td>
</tr>
<tr>
<td>Fe-Ni</td>
<td>131</td>
<td>190</td>
<td>570</td>
<td>2.12</td>
</tr>
</tbody>
</table>

Figure 5. Pore shape versus pore size relationship in both alloys, indicating that pores in the Cu-containing alloy were more rounded.

Figure 6. Tensile behavior of Fe-Ni-Cu and Fe-Ni alloys. Note the higher strength and work hardening rate in the Fe-Ni-Cu alloy.
The fatigue behavior for both alloys was also quite different. The stress versus cycles behavior for both alloys, Fig. 8, showed that the addition of Cu significantly increased fatigue resistance. In order to compare the fatigue strength (taken as fatigue runout at $10^7$ cycles) to tensile behavior, we examined the ratio of fatigue strength to tensile strength, $\sigma_{fat}/\sigma_{uts}$, and the ratio of fatigue strength to proportional limit stress, $\sigma_{fat}/\sigma_{pl}$, Table 4. Due to the porosity in these materials, the fatigue ratio of $\sigma_{fat}/\sigma_{uts}$ was significantly lower than for fully dense Cu-containing steels, where the ratio is between 0.5-0.6 [10]. While the $\sigma_{fat}/\sigma_{uts}$ ratio was around 0.25 for both alloys, the $\sigma_{fat}/\sigma_{pl}$ ratio was around 0.80 for both alloys. This can be explained by the fact the proportional limit stress is a very good indicator of the onset of plasticity, which in sintered materials takes place at the tips of the pores. In fatigue loading, this localized damage is more likely to be controlling fatigue life, than large-scale plasticity that takes place close to the ultimate tensile strength. Thus, the proportional limit stress may be a much better indicator of the fatigue strength than ultimate tensile strength. It should be noted that the method by which proportional limit is measured is limited by the accuracy of the technique and resolution of the stress-strain data acquisition, so more effective measurement techniques of the proportional limit stress may yield even better correlation to fatigue strength.

Figure 7. Tensile fracture surface, characteristic of both alloys, shown localized ruptured at sintered bonds.

Figure 8. Stress versus cycles behavior of Fe-Ni-Cu and Fe-Ni alloys. The addition of Cu enhances the fatigue resistance of the alloy.
Fractography indicated that fatigue crack initiation took place at large pores, particularly at the surface of the specimen, Fig. 9. Porosity has long been attributed as the preferred site for fatigue crack initiation at or near the surface in sintered materials [1-4, 11-16]. Polasik et al. [4] have also shown that in the high cycle fatigue regime, most of the fatigue life is spent in crack nucleation rather than crack propagation. Thus, while the Cu-containing alloy had a larger density of small secondary pores, which would be conducive to easier crack propagation since the mean free path between pores decreases, this did not significantly deter the fatigue strength. Rather, since the primary pore distribution in both alloys was not significantly different, the higher strength and fatigue resistance of the Fe-Ni-Cu matrix around the pores was more significant in contributing to the higher fatigue strength.

![Figure 9](image)

**Figure 9.** Surface pore associated with fatigue crack nucleation site.

The fatigue fracture surface also exhibited localized fatigue striations. Unlike striations in wrought materials, however, the striations in porous sintered materials seemed to be highly localized. This may be due to the multiple sites of favorable orientation and size, with respect to the loading axis, that underwent blunting and sharpening and give the step-like appearance of the striations. Similar localized fatigue striations were also observed by Rodzinak and Slesar [17].

![Figure 10](image)

**Figure 10.** Localized fatigue striations observed in both alloys.
CONCLUSIONS

The mechanical behavior of binder-treated Fe-0.85Mo prealloy with Cu, Ni, and graphite additions was investigated. In particular, the effect of Cu on tensile and fatigue behavior was studied, yielding the following conclusions:

1. The transient liquid phase formed by the presence of Cu resulted in more rounded pores in the Fe-Ni-Cu alloy compared to Fe-Ni. While the total porosity was similar in both alloys, a larger fraction of secondary pores was present in the Fe-Ni-Cu.

2. The addition of Cu resulted in an increase in proportional limit stress and ultimate tensile strength over the Cu-free alloy. Copper diffusion into iron seems to contribute to enhanced resistance to dislocation motion resulting in an increase in work hardening rate over the Fe-Ni alloy. Both alloys exhibited similar fracture mechanisms, consisting primarily of ductile rupture at sintered particle bonds.

3. Fe-Ni-Cu alloy had a higher fatigue resistance than the Fe-Ni alloy. The higher fatigue resistance can be attributed to the enhanced solid solution strengthening and more rounded pores provided by Cu in Fe, particularly since the amount of primary porosity in both materials was similar. The proportional limit stress appears to be a good indicator of fatigue strength, since it quantifies the onset of localized plasticity in these materials.

ACKNOWLEDGMENTS

The authors would like to thank Hoeganaes Corp. for financial support of this work.

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