Effects of Sintering Conditions on the Mechanical Properties of Metal Injection Molded 316L Stainless Steel

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In this work, tensile and fatigue properties of the metal injection molded 316L stainless steel were studied with the variation of sintering conditions. It was found that increasing sintering temperature and time resulted in a decrease in the porosity along with an increase in the grain size. With decreasing porosity, tensile strength and elongation increased, while yield strength maintained constant, which was attributed to the balanced effects of decrease of porosity and grain coarsening. Tensile strength and elongation were found to be largely dependent on the porosity rather than on the grain size since the fracture occurred mainly by microvoid growth and coalescence. A quantitative analysis on the yield strength was tried by considering the classical Hall–Petch equation and mechanistic models for yield strength of porous materials. The fatigue crack growth resistance at low ΔK level was not largely decreased when the porosity was increased up to 8%, but was significantly decreased when the porosity was increased to 17%. It was discussed in relation to the intrinsic nature of pore morphology and the extrinsic nature of crack closure.

KEY WORDS: metal injection molding; stainless steel; sintering; fatigue crack growth resistance; Hall–Petch equation.

1. Introduction

A metal injection molding (MIM) method is a relatively new technology to produce net shape parts in the powder metallurgy industry. Increased interest over recent years has been focused on this process, which has emerged as a cost effective and innovative process as compared to the traditional net shaping processes such as precision casting, forging and automated machining.1–3) Unlike the conventional powder metallurgy, the MIM process is near free of component design and shape constraints, and offers considerable scope for extending the powder processing technology to produce more complex and precise parts.4,5) There are four basic operations for the MIM process: i) formation of a powder-binder mixture as feedstock, ii) shaping the feedstock in an injection molding machine, iii) extraction of the polymer binder, and iv) sintering at high temperature.6) Precise control of these four steps is essential to obtain high-density materials with enhanced mechanical properties.7)

However, up to now, most of the related studies have been focused only on its binder system, sintering parameters and alloying technique for the production of near full density materials.8–11) For a wide application of the metal injection molding techniques, it is necessary to investigate microstructures and mechanical properties in relation to the above process variables.12–15) Therefore, the aim of the present study is to investigate the microstructural changes and corresponding tensile and fatigue properties of metal injection molded 316L stainless steel with the variation of sintering conditions (temperature and time), and to discuss the roles of porosity and grain size on the mechanical properties.

2. Experimental Procedures

The powders used in this study were the 316L stainless steel powders (about 10.8 μm mean particle size), which were produced in the ANVAL Co. Sweden by gas atomizing method.16) Table 1 and Fig. 1 show the chemical composition and the morphology of powders, respectively. The powders were spherical shape with the density of 7.96 Mg/m³. The mixing of powders with the binder (paraffin wax based) was carried out in the kneader at 150°C for 3.6 ks with the powder/binder ratio of 65/35 in volume percent. Then, specimens with a plate shape (44×110×6 mm) were injection molded under the pressure of 22 MPa. After the injection molding, plate specimens were exposed in the solvent at 45°C for 15×3.6 ks to eliminate the binder over 95%,17) and then, thermal debinding process18) was carried

<table>
<thead>
<tr>
<th>Type</th>
<th>Cr</th>
<th>Ni</th>
<th>Si</th>
<th>Mn</th>
<th>Cu</th>
<th>Mo</th>
<th>P</th>
<th>S</th>
<th>C</th>
<th>O</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L</td>
<td>16.3</td>
<td>10.3</td>
<td>0.53</td>
<td>1.50</td>
<td>0.28</td>
<td>2.26</td>
<td>0.017</td>
<td>0.005</td>
<td>0.025</td>
<td>0.09</td>
<td>Bal.</td>
</tr>
</tbody>
</table>
out in H$_2$ atmosphere following the heating route of Fig. 2(a).

Sintering process was also performed in H$_2$ atmosphere. By varying the sintering temperature (1 100°C, 1 320°C, 1 350°C) and time (1, 2, and 4×3.6 ks) at 1 350°C as shown in Fig. 2(b), several different microstructures were obtained, and analyzed with optical and scanning electron microscopy. Microstructures were clearly observed by electroetching with nital etchant (HNO$_3$ 50% /H$_2$O 50%) at a condition of 1.5 V for 90 sec. After the sintering process, shrinkage took place considerably throughout the specimen, but the shrinkage ratios were not much different in all directions.

Tensile test was carried out using an INSTRON 8501 testing machine at a strain rate of 3.8×$10^{-3}$/sec. The gauge dimension of tensile specimen was 4 mm×6 mm×22 mm. Fatigue test was also performed using compact tension (CT) type specimens as shown in Fig. 3 with a sinusoidal waveform at 20 Hz and R50.1. A pre-crack was made by load-shedding method in accordance with ASTM E647-86. Fatigue crack length was measured by the crack-opening displacement (COD) method and also by the traveling microscope with an accuracy of 10 mm. During the crack propagation, the crack closure loads were intermittently measured by the compliance curve method to calculate the effective stress intensity factor range, $\Delta K_{eff}$ ($= K_{max} - K_{cl}$). The crack closure load was determined as the load at the intersecting point between two straight lines, which were extrapolated from the linear sections of the compliance curve.19–21)

3. Results and Discussion

3.1. Microstructures and Tensile Properties

Figure 4 shows the microstructures sintered at different conditions. It is clearly shown that the porosity decreases with increasing sintering temperature and time. It is also noticed that significant grain growth was accompanied with the decrease of porosity. The detailed microstructural characteristics of each microstructure are represented in Table 2. The notations of 17P, 8P, 5P, 2P, 0.7P indicate that each specimen contains porosity of 17%, 8%, 5%, 2%, 0.7% in

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volume fraction, respectively. Here, the relative density was calculated from the density of raw powder (i.e., full density) 7.96 Mg/m³. It is also important to note in Fig. 4 that the morphology of pores was changed with the decrease of porosity. The pores were isolated either in the grain interior or at the grain boundaries in the specimens containing porosity up to 8%. However, when the porosity increased further to 17% (Fig. 4(a)), most of pores were connected to each other, consisting of notches at the grain boundaries.

Figure 5 shows the variation of tensile strength, yield strength, and elongation of the specimens containing various amount of porosity. It is interesting to note that yield strength maintained constant with the variation of the porosity, while tensile strength and elongation increased with the decrease of porosity. In general, yield strength, tensile strength and elongation of sintered materials should increase with the decrease of porosity. But in this study, yield strength was kept constant, which was presumably due to the balanced effects of the decrement of porosity and the grain growth. Decrease of porosity normally leads to the increase of yield strength, which is, then, suppressed by the grain growth phenomenon occurring at the same time.

In order to explain our tensile result, especially regarding to the yield strength (σy), both the Hall–Petch equation for grain size effect and the yield function applicable to the porous materials were considered in this study. Earlier work of Norstrom showed that the calculated yield strength of 316L stainless steel (wrought material) is given by,

![Fig. 4. SEM photographs showing sintered microstructures at different conditions: (a) 17P (1100°C, 1 h), (b) 8P (1320°C, 1 h), (c) 5P (1350°C, 1 h), (d) 2P (1350°C, 2 h) and (e) 0.7P (1350°C, 4 h). Here, P denotes porosity (vol%) of a specimen.](image)

![Fig. 5. Tensile properties of metal injection molded (MIMed) 316L stainless steels containing different amounts of porosity.](image)
\[ \sigma_y = 155 + 7.78D^{-1/2} \] ..........................(1)

In Eq. (1), only the effect of grain size on yield strength was considered. To account for the effect of porosity, the following plasticity relations for porous materials proposed by other investigators were considered.

### Plasticity Relations

\[ J_1^2 = \left( \frac{1}{3} \right) \left[ (1 - \phi^2) + (1 - \phi_{cr}^2) \right] \sigma_{y(m)}^2 \]

Gurson ..................................(2)

\[ J_1^2 = \frac{3}{4} \left( \frac{1 - \phi^2}{(3 - 2\phi^{1/4}) \ln \phi} \right) \sigma_{y(m)}^2 \]

Green ..................................(3)

\[ J_1^2 = \frac{2.49y^2}{27} - \frac{y^{0.28} J_2}{\phi} = \frac{1}{3} \sigma_{y(m)}^2 \]

Shima and Oyane.............................(4)

\[ J_1^2 = \left[ \frac{1 - \phi^2}{1 - (1 - \phi_{cr}^2)} \right] \sigma_{y(m)}^2 \]

Doraivelu.................................(5)

where, \( \sigma_{y(m)} \): yield strength of matrix with no porosity

\( \phi \): critical porosity of materials having virtual strength

\( J_1 \): hydrostatic stress \( (J_1 = \sigma_{11} + \sigma_{22} + \sigma_{33}) \)

\( J_2 \): the second invariant of deviatoric stresses

\[ J_1^2 = \frac{1}{6} [ (\sigma_{11} - \sigma_{22})^2 + (\sigma_{22} - \sigma_{33})^2 + (\sigma_{33} - \sigma_{11})^2 ] \]

\[ + \sigma_{12}^2 + \sigma_{23}^2 + \sigma_{31}^2 \]

For the uniaxial tensile test, \( \sigma_y \neq 0 \) and \( \sigma_t = 0 \). Using Taylor series expansion in the Eq. (2) and \( \phi = 0.293^{26} \) in the Eq. (5), the above equations are simplified as follows.

\[ \sigma_{y(m)} = \left( \frac{1 - \phi^2}{1 + \phi/4} \right) \sigma_{y(m)} \] Gurson ...............(2')

\[ \sigma_{y(sp)} = \left( \frac{3(1 - \phi_{cr}^{1/3})^2}{3 - 2\phi^{1/4}} \right) \sigma_{y(m)} \] Green .............(3')

\[ \sigma_{y(sp)} = \sqrt{3(1 - \phi^2)^{1/3} / 3 + 2.07\phi^{1/4}} \sigma_{y(m)} \] Shima and Oyane .......(4')

\[ \sigma_{y(sp)} = \sqrt{2(1 - \phi^2 - 1} \sigma_{y(m)} \] Doraivelu ...........(5')

where, \( \sigma_{y(sp)} \): yield strength of sintered material

\( \sigma_{y(m)} \): yield strength of fully dense material

The above equations indicate that the yield strength of sintered materials depends not only on the yield strength of matrix but also on the porosity form factor \( F(\phi) \).

When substituting \( \sigma_{y(m)} \) with \( \sigma_y \) in Eq. (1), the above four equations are in the form of Eq. (6).

\[ \sigma_{y(sp)} = F(\phi)(155 + 7.78D^{-1/2}) \] ..............(6)

With the variation of porosity \( \phi \) and grain size \( D \), the yield stresses were calculated and compared with the experimental results as shown in Fig. 6. Figure 6 clearly evidences that our experimental data are in good agreement with the calculated ones using Gurson’s porosity form factor.

\[ \sigma_{y(sp)} = \left( \frac{1 - \phi^2}{1 + \phi/4} \right)(155 + 7.78D^{-1/2}) \] ..............(7)

While the yield strength is greatly affected by both porosity and grain size, the effect of grain size on the tensile strength and elongation in porous material is found not significant in this study, which is relevant to the earlier study by Lindstedet et al.\(^{27,28}\) They have reported that tensile strength of sintered 316L steel is largely varied by the porosity and inclusions, but not by the grain size. Since the amounts of oxide inclusions are found to be similar in all specimens, the porosity is considered an important factor in affecting the tensile strength and elongation. From the results of this study, it is considered that the yielding where a small amount of plastic deformation is imposed is largely affected by two factors, grain size and porosity. However, when a large amount of plastic deformation is given, the role of grain size on elongation to fracture (or tensile strength) diminishes since the fracture would be typically controlled by the microvoid growth and coalescence.

### 3.2. Fatigue Crack Growth Rates

Figure 7 shows the fatigue crack growth rates (FCGR) of specimens containing various amounts of porosity. The wrought material (0% porosity) having an identical grain size \( (90 \mu m) \) as that of 0.7\% specimen was also tested for the comparison. It is shown that the FCGR progressively increases with the increase of porosity. It is interesting to note that all specimens containing 0.7–8% pores represented slightly higher fatigue crack growth rates than that of the wrought material, while the 17\% specimen showed consid-
In general, the nominal stress intensity factor range ($K_{\text{eff}}$) is largely influenced by the porosity, while the extrinsic effects are closely related to the grain size as well as the porosity. The crack closure is known to occur by premature contact between the faces of the crack, which is mainly attributed to (i) residual plastic stretch at crack wake, (ii) the oxide layers formed within a fatigue crack and (iii) microscopic roughness of the fatigue fracture surfaces. Among the above three factors, microscopic roughness (iii) is considered as a major factor to control the crack closure of a less porous material, though of its grain size, is lower than that of higher porosity material. While the contribution of grain size on the crack closure will not be significant, the lack of intense slip bands in the crack wake indicates that our material undergoes homogeneous deformation with multiple slip at the crack tip, which leads to the straight crack advance even in the coarse grained material. While the contribution of grain size on the crack closure is minimal, fracture surface roughness becomes largely influenced by another important microstructural factor, porosity here, since the crack will propagate in most cases following the links between the pores ahead of the crack tip. As a result, the crack closure of a less porous material, though of its larger grain size, is lower than that of higher porosity material.

When the closure stress intensity factor is subtracted from the nominal $K$, the $da/dN$ can be plotted in terms of the effective stress intensity factor range ($\Delta K_{\text{eff}}$), which is shown in Fig. 11. The trend of FCGR curves with $\Delta K_{\text{eff}}$ is very close to that of FCGR curves with $K_{\text{eff}}$ (Fig. 7), indicating that only the 17P specimen represents considerably lower intrinsic fatigue resistance than any other specimens. This clearly evidences that the extrinsic crack closure effect is not significant in the specimens of 0.7P–8P and the FCGR is mainly governed by the intrinsic nature, which is greatly influenced by the porosity of the specimens.

The fatigue crack propagation resistance at early stage of
crack growth can be quantified by the near threshold stress intensity factor range (ΔK_{near}), which is typically determined when the fatigue crack growth rate approaches to 10^{-10} m/cycle. The ΔK_{near} is also composed of two terms reflecting intrinsic and extrinsic nature, respectively, as shown below.

\[ \Delta K_{\text{near}} = \Delta K_{\text{near, eff}} + K_{\text{near, cl}} \]  \hspace{1cm} (9)
The values of above terms can be obtained using the data in Fig. 7 and Fig. 11, and are represented in Table 4 and Fig. 12, respectively. It is clearly shown that increasing the porosity, the intrinsic fatigue resistance at near threshold region \((\Delta K_{\text{near th, eff}})\) decreases slowly up to the porosity level of 8%, and then decreases rapidly afterwards. In contrast, the extrinsic fatigue resistance \((K_{\text{near th, cl}})\) represents a reverse trend. As a result, the overall fatigue resistance at near threshold region \((\Delta K_{\text{near th}})\) decreases slightly with the increase of porosity to 8%, but decreases significantly when the porosity exceeds about 10%. It is thought that a sudden decrease of \(\Delta K_{\text{near th}}\) in 17P specimen is mainly attributed to the deleterious pore morphology of the microstructure (Fig. 4(a)), i.e., wedge type cracks or cavities interconnected to each other at grain boundaries. It is noted that if the pores are trapped in the grain interior or at the grain boundary maintaining their spherical shape, the decrease of \(\Delta K_{\text{near th}}\) is not significant. The importance of pore morphology on the fatigue resistance is further evidenced from the fatigue test results of the two specimens containing identical porosity but with different pore morphology. Figures 13(a) and 13(b) show the microstructures obtained by conventional powder metallurgy (P/M) and metal injection molded process, respectively. Although two microstructures have similar porosity (8%) and grain sizes (45–50 \(\mu m\)), the \(\Delta K_{\text{near th}}\) of conventional PM processed specimen is considerably lower than that of MIMed specimen (Fig. 14), owing to its wedge type morphology of pores. Therefore, the pore morphology as well as porosity is considered important in affecting the mechanical proper-

### Table 4

<table>
<thead>
<tr>
<th>Porosity (%)</th>
<th>17P</th>
<th>8P</th>
<th>5P</th>
<th>2P</th>
<th>0.7P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength (MPa)</td>
<td>260</td>
<td>450</td>
<td>489</td>
<td>506</td>
<td>531</td>
</tr>
<tr>
<td>Yield strength (MPa)</td>
<td>193</td>
<td>173</td>
<td>177</td>
<td>178</td>
<td>182</td>
</tr>
<tr>
<td>Grain size ((\mu m))</td>
<td>12</td>
<td>45</td>
<td>63</td>
<td>77</td>
<td>90</td>
</tr>
<tr>
<td>Elongation (%)</td>
<td>6</td>
<td>29</td>
<td>43</td>
<td>50</td>
<td>61</td>
</tr>
</tbody>
</table>

The data in Table 4 and Fig. 12 show that increasing the porosity, the intrinsic fatigue resistance at near threshold region \((\Delta K_{\text{near th, eff}})\) decreases slowly up to the porosity level of 8%, and then decreases rapidly afterwards. In contrast, the extrinsic fatigue resistance \((K_{\text{near th, cl}})\) represents a reverse trend. As a result, the overall fatigue resistance at near threshold region \((\Delta K_{\text{near th}})\) decreases slightly with the increase of porosity to 8%, but decreases significantly when the porosity exceeds about 10%. It is thought that a sudden decrease of \(\Delta K_{\text{near th}}\) in 17P specimen is mainly attributed to the deleterious pore morphology of the microstructure (Fig. 4(a)), i.e., wedge type cracks or cavities interconnected to each other at grain boundaries. It is noted that if the pores are trapped in the grain interior or at the grain boundary maintaining their spherical shape, the decrease of \(\Delta K_{\text{near th}}\) is not significant. The importance of pore morphology on the fatigue resistance is further evidenced from the fatigue test results of the two specimens containing identical porosity but with different pore morphology. Figures 13(a) and 13(b) show the microstructures obtained by conventional powder metallurgy (P/M) and metal injection molded process, respectively. Although two microstructures have similar porosity (8%) and grain sizes (45–50 \(\mu m\)), the \(\Delta K_{\text{near th}}\) of conventional PM processed specimen is considerably lower than that of MIMed specimen (Fig. 14), owing to its wedge type morphology of pores. Therefore, the pore morphology as well as porosity is considered important in affecting the mechanical proper-
ties, especially the fatigue resistance, of MIMed specimens.

5. Conclusions

The present investigation was made to study the tensile and fatigue properties of metal injection molded 316L stainless steels with the variation of sintering conditions. Important conclusions drawn from this work are as follows.

1) With increasing the sintering temperature and time, the porosity of sintered specimen was decreased accompanying the simultaneous grain growth.

2) Tensile strength and elongation were found to be largely dependent on the porosity, while yield strength was not significantly varied. This insensitive variation of yield strength with the porosity was mainly attributed to the balanced effects of the decrease of porosity and concurrent grain coarsening.

3) A quantitative analysis for yield strength as functions of grain size and porosity was made by considering the classical Hall–Petch equation and mechanistic models for yield strength of porous materials. The experimental data of present study were in good agreement with the equation, which utilized the Hall–Petch equation and Gurson’s plasticity relation for porous materials.

4) The fatigue crack growth resistance at low $\Delta K$ regime ($\Delta K_{\text{near th}}$) was analyzed in view of the intrinsic nature of pore morphology and the extrinsic nature of crack closure. Up to the porosity level of 8% the $\Delta K_{\text{near th}}$ was not largely decreased, which was attributed to the beneficial (trapped and isolated) pore morphology.

REFERENCES