Fatigue Strength and Fracture Surface Morphology in Ultra-fine Grained Ferritic Stainless Steels

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Abstract: Ferritic stainless steels such as Fe-2.25Cr-1Mo-1Zr, Fe-25Cr-1Zr and Fe-13Cr-8Ni-2Mo-1Zr with ultra-fine grain size of 0.2 µm were prepared by hot extrusion after mechanical alloying. Fatigue strength expressed as maximum stress at 3x10^6 cycles for Fe-2.25Cr-1Mo-1Zr, Fe-13Cr-8Ni-2Mo-1Zr and Fe-25Cr-1Zr were 1150, 1150 and 1350 MPa, respectively. Transgranular fracture surfaces were predominantly observed at crack initiation and crack propagation area. Striation like pattern was observed at a restricted crack propagation area. The mechanism on higher fatigue strength in ultra-fine grained materials is discussed in terms of fracture surface morphology on the basis of the recent investigations. It can be concluded that the higher fatigue strength of ultra-fine grained ferritic stainless steels is strongly governed by delay of crack initiation.

1. Introduction

It is well recognized that grain refinement increase ultimate tensile strength of metallic materials. Grain refinement also increase fatigue strength of metallic materials[1-7]. However fatigue strength and fatigue behavior of steel with ultra-fine grain size lower than 1 µm have not clarified yet. So far materials with grain size of few micron meter were produced by conventional methods such as heat treatment, thermo mechanical processing and strong working at the phase transition points. Recent manufacturing process with mechanical alloying and hot extrusion enabled to manufacture materials with ultra-fine grain size of lower than 1 µm. In this paper fatigue strength and fracture surface morphology of ferritic stainless steels with ultra-fine grain size of 0.2 µm made by mechanical alloying and hot extrusion process are presented.

2. Experimental procedures

2.1 Materials

The ultra-fine grained ferritic stainless steels with 26mm diameter was extruded after mechanical alloying. The mechanical alloying condition is summarized in Table 1. The extrusion temperature was 1023 to 1123K and the ratio of extrusion was 6.0. The prepared ferritic stainless steels were Fe-2.25Cr-1Mo-1Zr, Fe-25Cr-1Zr and Fe-13Cr-8Ni-2Mo-1Zr. In this process ultra-fine grained structure was formulated by numerous recrystallized nuclei with internal strain. The additive Zr prevent grain coarsening during mechanical alloying by pinning action at grain
Table 1. MA process condition.

<table>
<thead>
<tr>
<th>MA Condition (Attrition Mill)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Materials</td>
<td>5kg, Particle size &lt;180 μm</td>
</tr>
<tr>
<td>Chamber</td>
<td>SUS304, Volume 25L</td>
</tr>
<tr>
<td>Ball</td>
<td>75kg, 3/8-inch SUJ2</td>
</tr>
<tr>
<td>Atmosphere</td>
<td>Ar, 30mL/min</td>
</tr>
<tr>
<td>Arm</td>
<td>250rpm (rotational frequency)</td>
</tr>
<tr>
<td>Treatment time</td>
<td>48Hr</td>
</tr>
</tbody>
</table>

Fig. 1 TEM microstructure of used steels.

a) Fe-2.25Cr-1Mo-1Zr
b) Fe-13Cr-8Ni-2Mo-1Zr
c) Fe-25Cr-1Zr
boundary. The TEM microstructure of these ferritic stainless steels are shown in Fig.1. The diameter of the grain size is about 0.2 μm. The grain size is affected by the temperature of extrusion. The representative chemical compositions of these ferritic stainless steels are shown in Table 2. Fatigue fracture surfaces were observed by scanning electron microscope (JEOL JSM-6480V).

Table2 Chemical compositions of used ferritic stainless steels (mass % ).

<table>
<thead>
<tr>
<th>Material</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Zr</th>
<th>N</th>
<th>O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-13Cr-8Ni-2Mo-Zr</td>
<td>0.17</td>
<td>0.03</td>
<td>0.10</td>
<td>0.005</td>
<td>0.003</td>
<td>12.8</td>
<td>7.89</td>
<td>2.23</td>
<td>0.90</td>
<td>0.016</td>
<td>0.074</td>
</tr>
<tr>
<td>Fe-25Cr-1Zr</td>
<td>0.13</td>
<td>0.02</td>
<td>0.11</td>
<td>0.004</td>
<td>0.002</td>
<td>25.0</td>
<td>-</td>
<td>-</td>
<td>0.94</td>
<td>0.010</td>
<td>0.078</td>
</tr>
<tr>
<td>Fe-2.25Cr-1Mo-Zr</td>
<td>0.043</td>
<td>0.03</td>
<td>0.10</td>
<td>0.004</td>
<td>0.002</td>
<td>2.41</td>
<td>-</td>
<td>1.00</td>
<td>0.95</td>
<td>0.016</td>
<td>0.12</td>
</tr>
</tbody>
</table>

Fig. 2 shows the relationship between ultimate tensile strength and elongation. The ultimate tensile strength is higher than that of conventional steel [8] and is smaller for Fe-25Cr-1Zr, Fe-2.25Cr-1Mo-1Zr and Fe-13Cr-8Ni-2Mo-1Zr in order. These ultra-fine grained steels have an excellent impact energy, and wear resistance [9].

2.2 Fatigue test
Fatigue tests were conducted by use of an Instron8502 (250kN) fatigue testing
machine. Testing frequency was 20Hz and R (the ratio of minimum to maximum loading ratio) was 0.1. Testing temperature was controlled to 473K during the test by use of a thermostatic chamber. The figure and the dimensions of the fatigue test specimen is shown in Fig.3.

![Fatigue test specimen](image)

Fig.3 Fatigue test specimen.

### 3. Results and discussion

#### 3.1 Fatigue life test

Fig.4 shows S-N diagrams of ultra-fine grained ferritic stainless steels. The fatigue strength of maximum stress at $3 \times 10^6$ cycles of Fe-2.25Cr-1Mo-1Zr, Fe-
13Cr-8Ni-2Mo-1Zr and Fe-25Cr-1Zr is 1150, 1150 and 1350 MPa, respectively. In this figure it is apparent that the fatigue strengths of these ferritic stainless steels are much higher than those of conventional steels of SAE8740 and SNB16. The ratio of the fatigue limit to ultimate tensile strength at room temperature of Fe-2.25Cr-1Mo-1Zr, Fe-13Cr-8Ni-2Mo-1Zr and Fe-25Cr-1Zr is 0.7, 0.64 and 0.68, respectively. These ratios are higher than those of conventional steels of 0.41 for SAE8740 and 0.49 for SNB16.

So far the fatigue resistance of the parts made by conventional powder metallurgy was recognized to be questionable because of the residual porosity[10]. Fatigue strength decrease due to early crack initiation from the porosity of materials made by powder metallurgy can be easily anticipated. One of the reason of the improvement of fatigue strength of these tested steels is that the ferritic stainless steels without micro porosity were made by the hot extrusion after mechanical alloying. Another reason is the ultra-fine grain size with 0.2 μm of these tested steels. Of course it has been recognized that grain refinement can improve the fatigue strength of stainless steels [11]. Recently the improvement of fatigue strength of various materials with nano meter grain size have been reported on steels (Grain size ; 1.8 to 4.5 μm)[2-4], Ti alloy (Grain size 0.2 to 0.5 μm)[1,6], aluminum alloy (Grain size; 0.2 to 0.5 μm)[5] and copper (Grain size 0.3 μm)[6]. One of the authors have reported that fatigue strength of the protium treated Ti-6Al-4V alloy with grain size of 0.2 μm showed 60 percent higher fatigue limit of conventional Ti-6Al-4V alloy with grain size of 5.7 μm. The reason of fatigue strength improvement can be attributed to delay of fatigue crack initiation in ultra fine grained Ti-6Al-4V alloy with higher resistance for fatigue crack initiation[6]. The improvement of fatigue strength of these ferritic stainless steels can be explained by the delay of fatigue crack initiation and transgranular mode induced by ultra-fine grain structure as after mentioned.

### 3.2 Fatigue fracture surface morphology

Fig.5, Fig.6 and Fig.7 shows the representative fatigue fracture surfaces of Fe-2.25Cr-1Mo-1Zr, Fe-13Cr-8Ni-2Mo-1Zr and Fe-25Cr-1Zr, respectively. In most of the specimen fatigue crack initiated at the surface and propagated with transgranular path [Fig.5b, Fig.6b and Fig.7b]. Fracture surfaces at the crack initiation area are characterized with transgranular fractures with ultrafine grains [Fig.5a, Fig.7a]). Subsurface crack initiation was observed for fracture surface of Fe-13Cr-8Ni-2Mo-1Zr specimen failed at higher number of cycles as shown in Fig.6a). While, crack initiated at the surface of Fe-13Cr-8Ni-2Mo-1Zr specimen failed at higher number of cycles. The subsurface crack initiation and the morphology changes at crack initiation area are most frequently observed on fracture surfaces in high strength steels such as cold forging die steels [12]. Fracture surfaces at early crack propagation area are also characterized with transgranular fractures with ultra-fine grains. Transgarnular fractures were also predominant at crack propagation area for these ferritic stainless steels. Secondary cracking [Fig.5c, Fig.7c] and striation like fracture surface [Fig.6b)] at restricted area were also observed at crack propagation area. The secondary cracking was predominantly observed at the fracture surface failed at higher repeated stress.
Fig. 5 Fatigue fracture surface of Fe-2.25Cr-1Mo-1Zr steel.
\( \sigma_{\text{max}} = 1150 \text{MPa}, \ N_f = 2.92 \times 10^5 \text{cycles} \\
\) a) initiation, b) 0.3mm from initiation ,c)0.8mm from initiation.
Fig. 6 Fatigue fracture surface of Fe-13Cr-8Ni-2Mo-1Zr steel.
\[ \sigma_{\text{max}} = 1095 \text{MPa}, N_f = 8.75 \times 10^5 \text{ cycles} \]
a) initiation, b) 0.5mm from initiation, c) 0.9mm from initiation.
Fig. 7 Fatigue fracture surface of Fe-25Cr-1Zr steel

\[ \sigma_{\text{max}} = 1388 \text{MPa}, \quad N_f = 1.09 \times 10^4 \text{ cycles} \]

a) initiation, b) 0.3mm from initiation, c) 0.7mm from initiation.
Thus fatigue fracture surfaces of these tested ferritic stainless steels are characterized with transgranular fracture mode at initiation and propagation area. Therefore the higher fatigue strength of these ferritic stainless steels is attributed to ultra-fine grain structure. Striation like pattern was also observed at restricted crack propagation area of protium treated Ti-6Al-4 V alloy. Striation like pattern can be clearly identified between transgranular fracture as shown in Fig.8. The protium treated Ti-6Al-4V alloy with ultra fine grain size of 0.2 µm showed the higher fatigue strength than that of conventional Ti-6Al-4V alloy[6]. It can be mentioned that these materials with high strength has a little brittleness. The phenomenon reported on high strength VT9 titanium alloy with ultimate tensile strength of 1350 MPa that brittle fracture surrounded by dimpled relief [13] can be mentioned to be the same as the striation like pattern observed on ferritic stainless steels and protium treated Ti-6Al-4V alloy with ultra-fine grain size.

Fig.8 Transgranular fracture surface and striation like pattern observed in crack propagation area of ultra-fine grained Ti-6Al-4V alloy.
Maximum stress ;500MPa [6]

4. Conclusions

1) Fatigue strength expressed as maximum stress at 3x10^6 cycles of ferritic stainless steels of Fe-2.25Cr-1Mo-1Zr,Fe13Cr-8Ni-2Mo-1Zr and Fe-25Cr-1Zr with ultra fine grain is 1150,1150 and 1350Mpa,respectively.

2) Transgranular fracture surfaces were predominant at fatigue crack initiation and propagation area. Striation like fracture surface was observed at restricted
crack propagation area.

References