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

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Abstract : Ferritc stainless steels such as Fe-2.25Cr-1Mo-1Zr,Fe-25Cr-1Zr and Fe-13Cr-8Ni-2Mo-1Zr with ultra-fine grain size of 0.2µm 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 μ m 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 μ m. In this paper fatigue strength and fracture surface morphology of ferritic stainless steels with ultra-fine grain size of 0.2 μ m 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 Table1. 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

Table1. MA process condition.

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

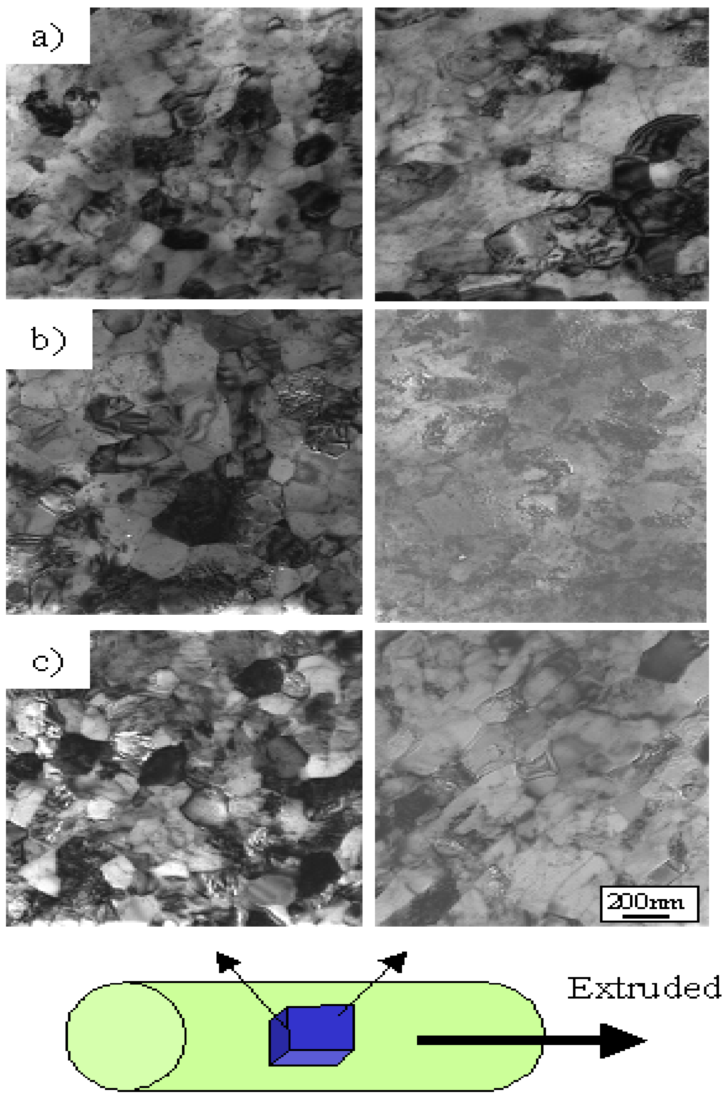

Fig.1 TEM microstructure of used steels.
a) Fe-2.25Cr-1Mo-1Zr
b) Fe-13Cr-8Ni-2Mo-1Zr
c) Fe-25Cr-1Zr

boundary. The TEM microstructure of these ferritic stainless steels are shown in Fig.1. The diameter of the grain size is about 0.2 μ m. 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 %).

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

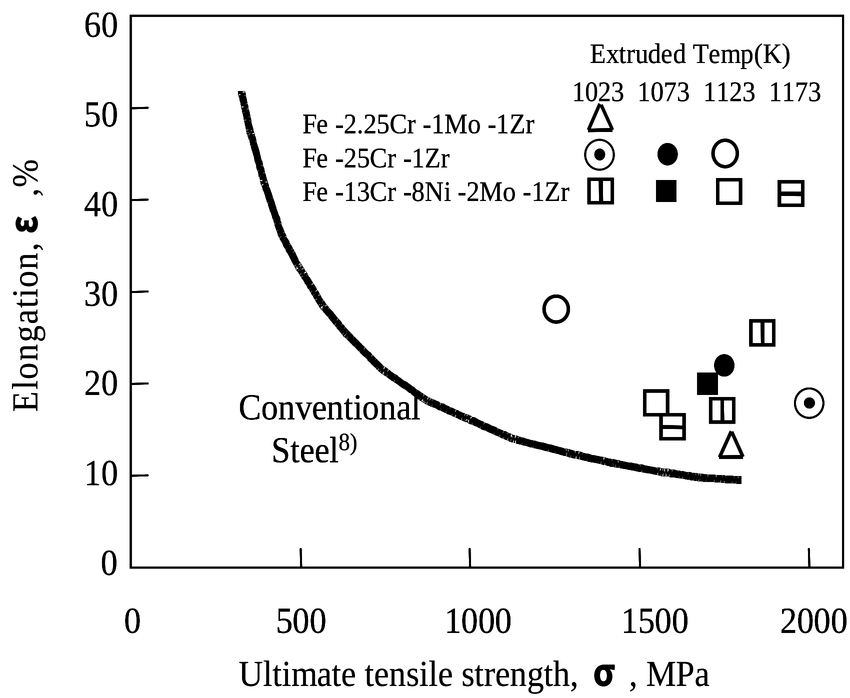

Fig. 2 Relationship between ultimate tensile strength and elongation of ultra-fine grained ferritic stainless steel.

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 473Kduring the test by use of a thermostatic chamber. The figure and the dimensions of the fatigue test specimen is shown in Fig.3.

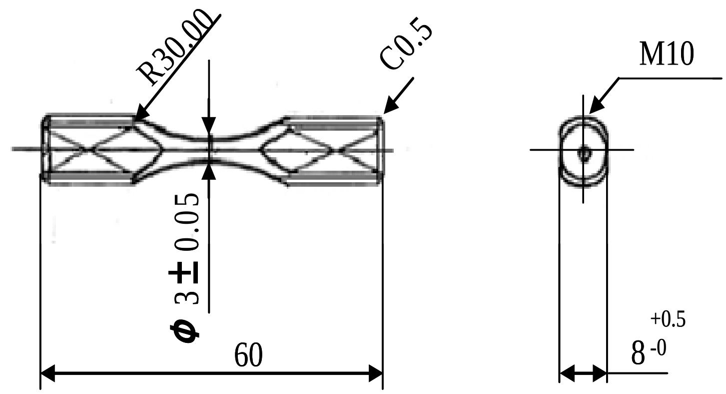

Fig.3 Fatigue test specimen.

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 $3x10^6$ cycles of Fe-2.25Cr-1Mo-1Zr, Fe-

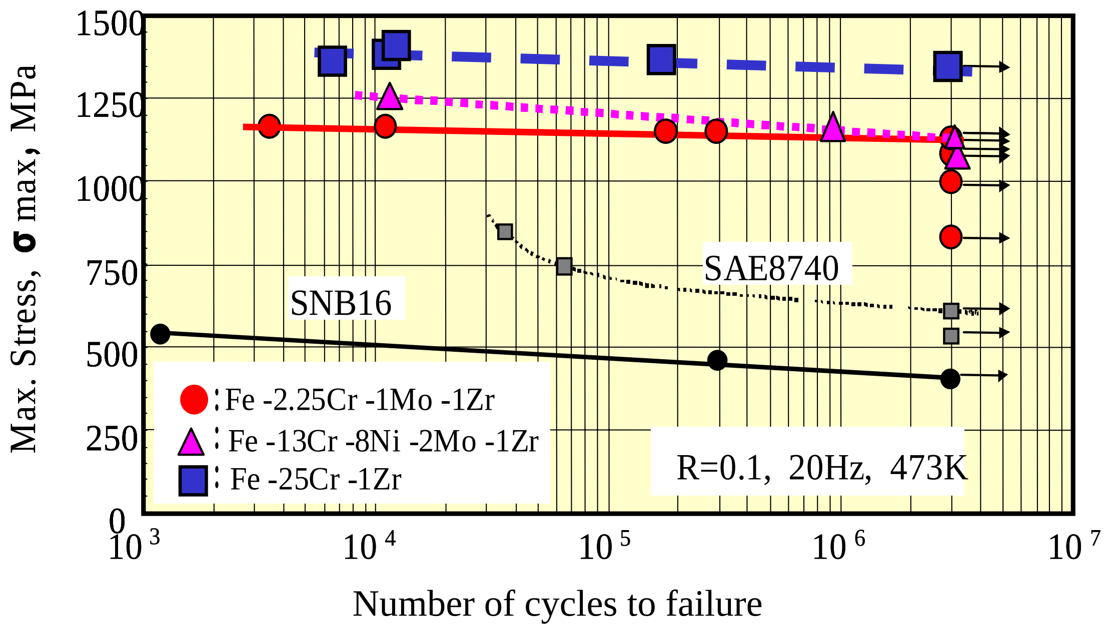

Fig.4 S-N diagrams of ultrafine grained ferritic stainless steels.

13Cr-8Ni-2Mo-1Zr and Fe-25Cr-1Zr is 1150,1150 and 1350MPa,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 repeared stress.

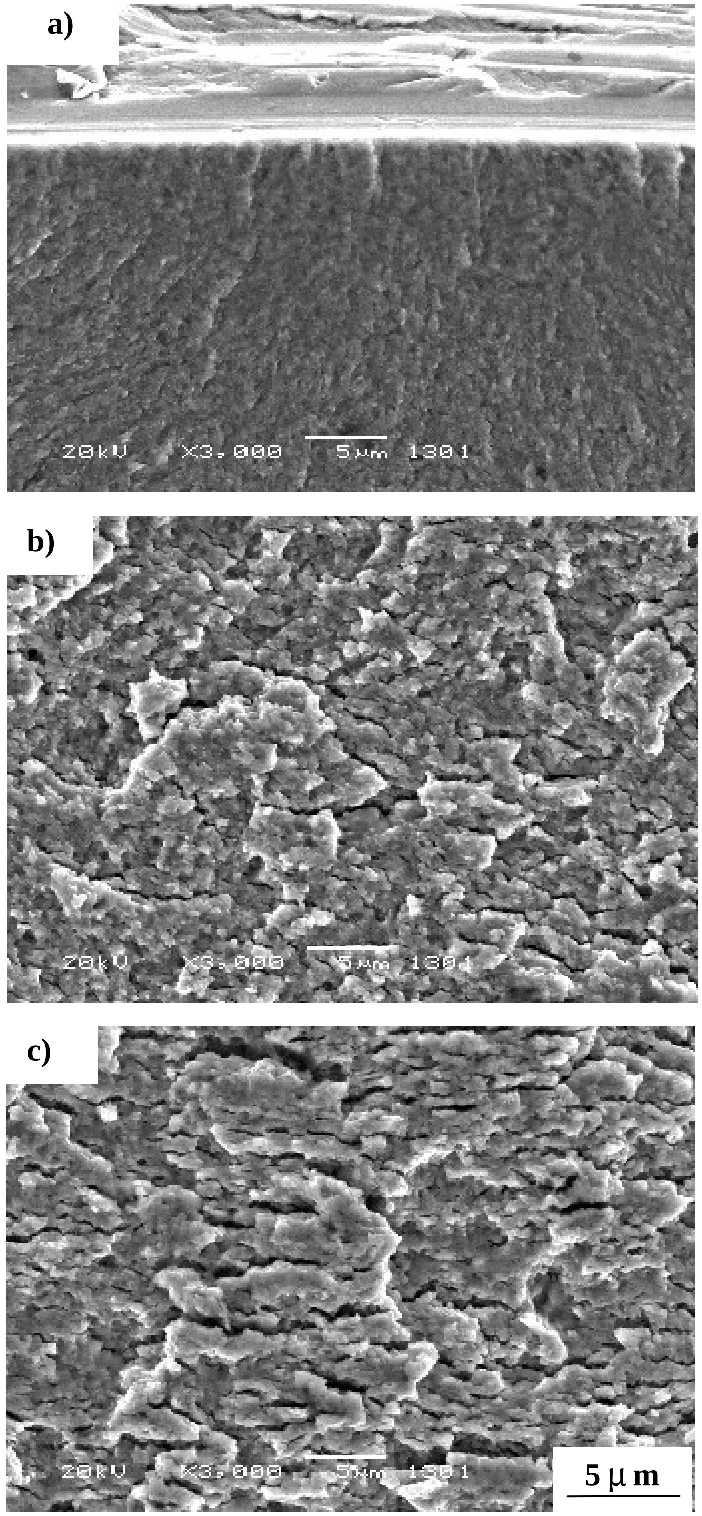

Fig.5 Fatigue fracture surface of Fe-2.25Cr-1Mo-1Zr steel.
σ_{max}=1150MPa、N_f=2.92x10^5cycles
a) initiation, b) 0.3mm from initiation ,c)0.8mm from initiation.

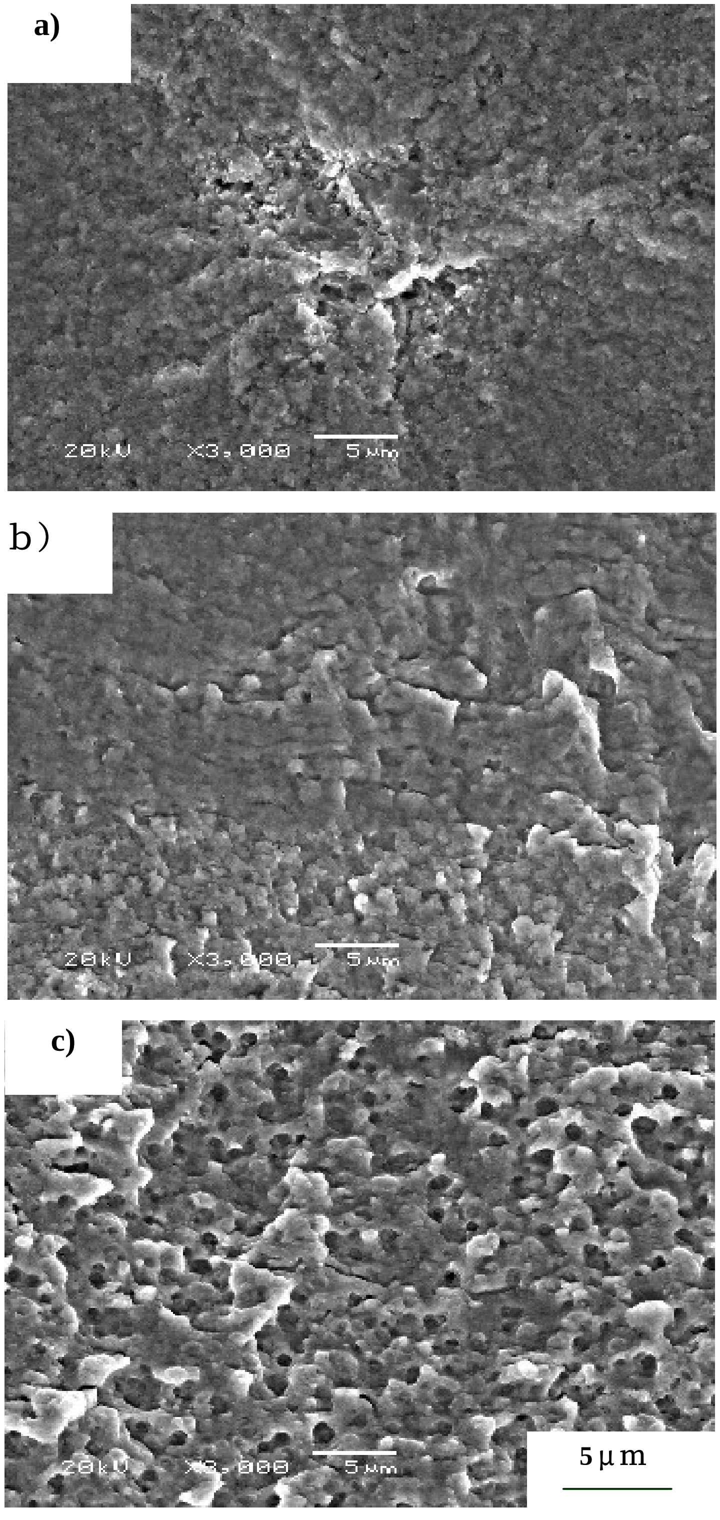

Fig.6 Fatigue fracture surface of Fe-13Cr-8Ni-2Mo-1Zr steel.
σ_{max}=1095MPa,N_f=8.75x10^5 cycles
a)initiation, b)0.5mm from initiation,c) 0.9mm from initiation.

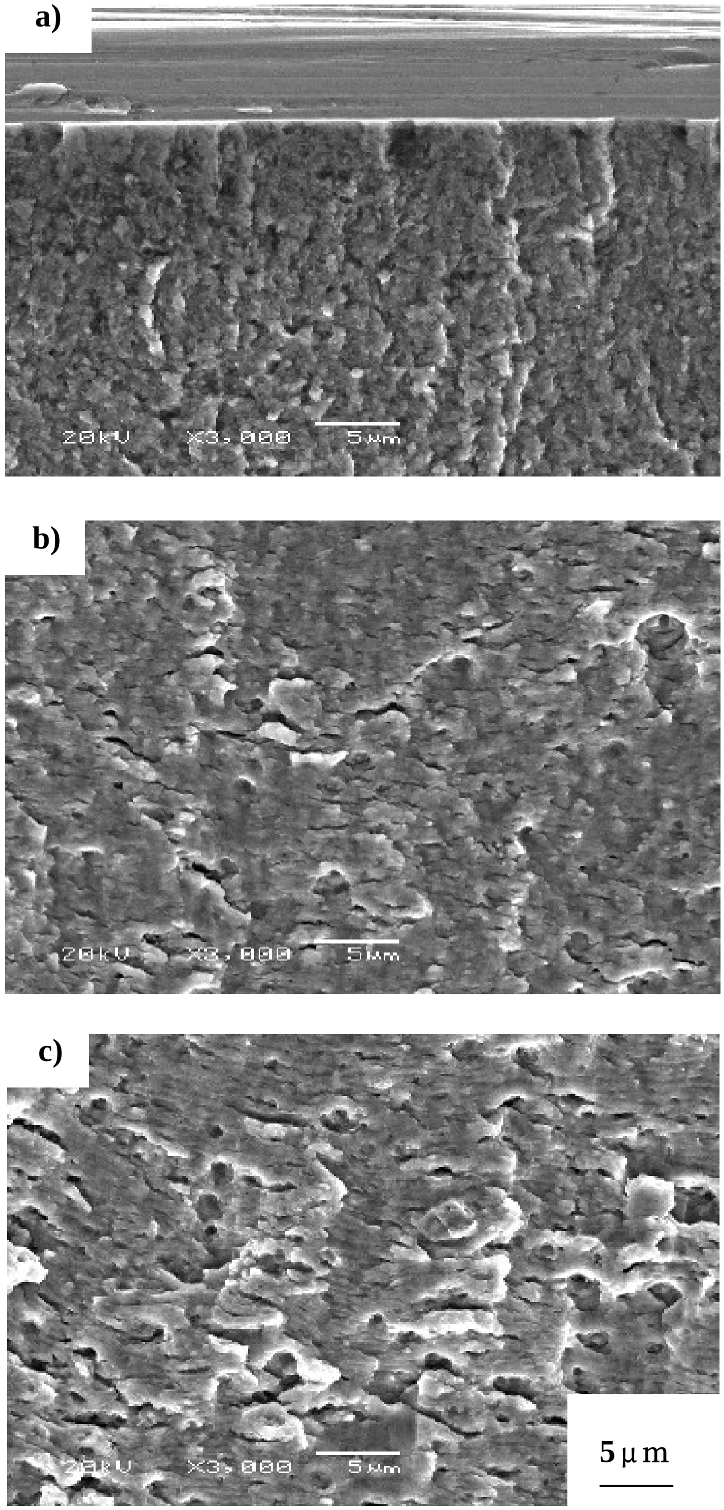

Fig.7 Fatigue fracture surface of Fe-25Cr-1Zr steel
σ_{max}=1388MPa、N_f=1.09x10^4 cycles
a) initiation, b) 0.3mm from initiation,c) 0.7mm from initiation.

Thus fatigue fracture surfaces of these tested ferritic stainless steels are characterized with trangranular 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 protioum treated Ti-6Al-4V alloy with ultra-fine grain size.

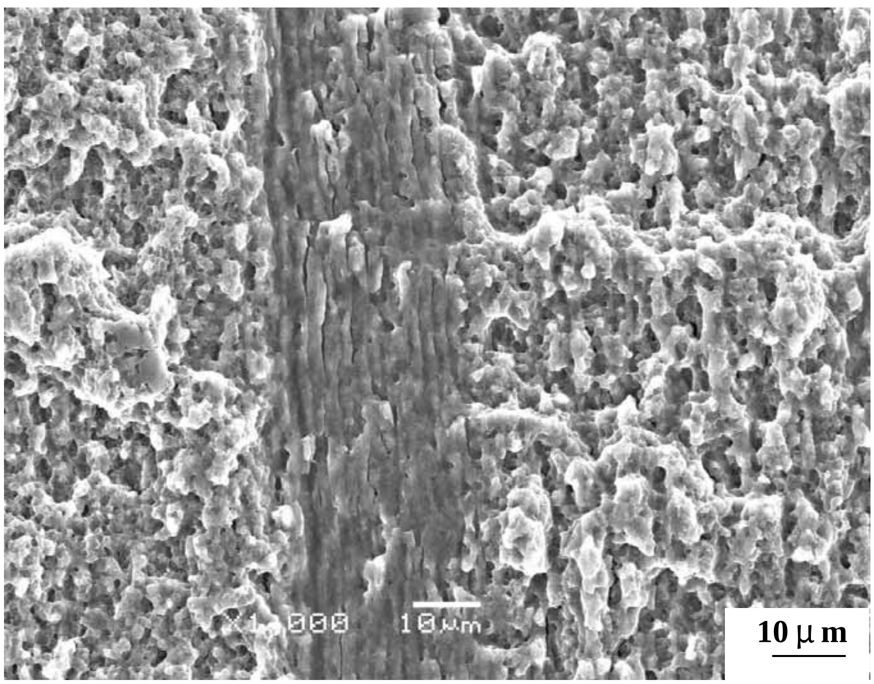

Fig.8 Transgranular fracture surface and striation like pattern observed in crack propagation area of ultra-fine grained Ti-6Al-4V alloy.
Maximum stress ;500MPa [6]

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

[1]H.Yoshimura,Y.Fujii,S.Wada,A.Murakami and K.Kimura, Tensile,impact and fatigue properties of ultra-fine graines $\alpha+\beta$ type Titanium alloys prepared by hydrogen treatment, Japan Inst. of Metals,Vol.61(1997),83-89

[2]H.Yajima,M.Yamamoto,T.Ishikawa and T.Fukui,Fatigue strength in sea water of steel plate having surface layers with ultra fine grain microstructure and its evaluation,Trans.of the West-Japan Soc. of Naval Architectures, No94(1997)183-193

[3] H.Kimura,Y.Akiniwa,K.Tanaka,J.Kondo and T.Ishikawa,Effect of microstructure on fatigue crack propagation behavior in ultrafine-grained steel ,J.Soc. of Materials Science,Japan, Vol.51(2002),796-800

[4]H.Kimura,Y.Akiniwa,K.Tanaka,J.Kondo and T.Ishikawa,Atomic force microscopic observation of initiation and early propagation of small fatigue cracks in ultrafine-grained steel, J.Soc.Materials Science,Japan,Vol.51 (2002),801-807

[5] J.Sugeta,Y.Uematsu,M.Yasuda and M.Jono,Initiation and growth behavior of small fatigue cracks in ultra-fine grained p/m aluminum alloy at elevated temperatures, J.Soc.of Materials Science,Japan, Vol.55(2006),545-549

[6]T.Nakaiso,M.Tanada,R.Ebara,J.Nakahigashi, H.Yoshimura and K.Yamamoto, Fatigue strength of ultra-fine grained Ti-6Al-4V Alloy" Proc.of the Fatigue (2006)CDROM

[7]Y.Kojima,H.Kimura,Y.Akiniwa,K.Tanaka and K.Tsuji,Fatigue crack initiation and propagation behavior of ECAP-processed Copper investigated by EBSD, Proc. Of AREM2007(CDROM)(2007).

[8]O.Hashimoto, Strengthening technology of thin steel in transportation apparatus, The Advantage of Strengthening Technology in Steel Products, #146,147 Nishiyama Memorial Lecture,ISIJ(1972) 119

[9]Y.Matsumura and K.Kishi,Invention and evaluation of steels with ultra-fine microstructure by powder metallurgy,Kinzoku,Vol.76(2006)557-563

[10]A.Lawley, Powder metallurgy for high performance application, Proc. of the 18th Sagamore Army Materials Research Conference, J.J.Burke and V. Weiss (Eds.), Syracuse Univ. Press,p3,(1972).

[11]R.C.Gibson and J.H.Brophy,Micro duplex Nickel-Iron-Chromium alloys,Ultrafine-grain metals,Proc. of the 16 th Sagamore Army Materials Research Conf., J.J.Burke and V.Weiss(eds) ,Syracuse Univ.Press(1970) pp.377-395

[12] T.Yamashita,T.Bito,R.Ebara and K.Kubota,Ion nitride effect on fatigue strength of hot forging die steels,Proc. of the ATEM2007,JSME(2007) CDROM.

[13]A.Shanyavskiy,T.Zaharova and Y.Potahenko,The nature of multi-modal distribution of fattigue durability for titanium alooy VT9,Proc.of the fourth International Conf.on Vey High Cycle Fatigue,J.E.Allison,W.Jones,M.Larsen and R.O.Ritchie(Eds),TMS(2006)325-330