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THE COMPARISON OF YIELD AND FATIGUE STRENGTH DEPENDENCE ON GRAIN SIZE OF PURE Ti PRODUCED BY SEVERE PLASTIC DEFORMATION

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Abstract. Microstructure evolution and the mechanical behavior of UFG commercially pure Ti (grade 2) produced by ECAP were investigated, using the procedure designated as route B_c. Repetitive pressings of the same sample were performed to 4 passes at 683K. After four pass, recrystallized gains were uniformly distributed throughout the specimen with an average size of 0.4 mm. The fatigue limit σ_e of the pure Ti increased by 29% from 183.1 to 236.1 MPa after 4 pass ECAP. This result indicates that a significant improvement in high-cycle fatigue life can be achieved by applying ECAP on pure Ti. The standard Hall-Petch relation for yield stress and fatigue limit exhibit in the ECAPed Ti. The grain size dependence of k_y and k_e in the present Ti are 7.5 MPa $\sqrt{\text{mm}}$ and 2.5 MPa $\sqrt{\text{mm}}$, respectively. The present Ti exhibits higher σ_o compared to σ_{oe}.

1. INTRODUCTION

Equal Channel Angular Pressing (ECAP) process has been recognized to be quite effective in improving the strength of many metallic alloys through (sub)grain refinement [1,2]. The ECAP process has been applied mainly to metals and alloys having cubic crystal structure, and the number of severe plastic deformation studies on hexagonal materials is limited. Utilizing ECAP for pure Ti is great interest for its enhancement in tensile and fatigue strength through grain refinement. However, the fatigue properties of the pure Ti produced by ECAP have not been systematically studied. The purpose of this study is to develop a better understanding of the effect on fatigue and yield strength of pure Ti produced by ECAP.

2. EXPERIMENTAL

A commercially pure titanium (grade 2) was cut to the rods with diameter of 14.5 mm and length of 90 mm and then annealed at 1073K for 1 hr in Ar atmosphere, and then quenched into room-temperature water. Its chemical composition was Ti-0.06Fe-0.01N-0.01O-0.01H (wt.%). The average grain size was 105 μm after the heat treatment. ECAP was conducted using a die with an internal angle Φ of 110° and an outer curvature angle Ψ of 25°. The present ECAP die was designed to give an approximate strain ε of ~0.76 on each pressing according to the following equation [2];

$$\varepsilon=\frac{2\cot\left(\Phi/2+\Psi/2\right)+\Psi\,\mathrm{cosec}\left(\Phi/2+\Psi/2\right)}{\sqrt{3}}. \quad (1)$$

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Fig. 1. The tensile and fatigue samples extracted from the ECAPed rod.

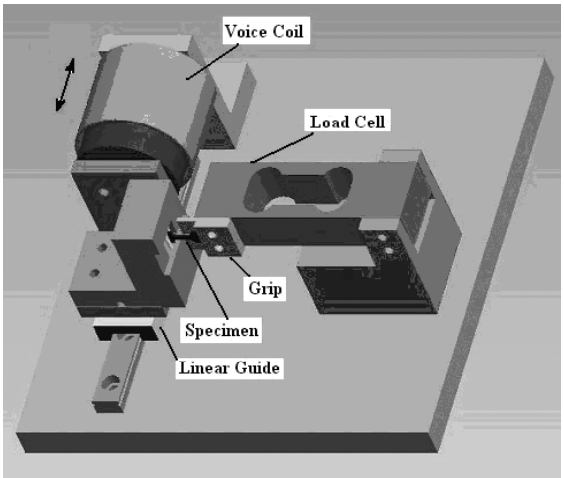

Fig. 2. Set-up of the developed fatigue testing machine.

Repetitive pressings of the same sample were performed to 4 passes. During ECAP, all pressings were conducted at 683K, using the procedure designated as route B_c, in which each sample was rotated 90° around its longitudinal axis between the passages. The details of the ECAP processing have been reported elsewhere [3]. Tensile properties in longitudinal directions of the ECAPed and unECAPed Ti rods were measured using the miniature tensile specimens with geometry of 3 mm gauge length, 1 mm gage width, 1 mm thickness, and 2 mm shoulder radius. The tensile and fatigue samples were extracted from the center portion of the ECAPed and unECAPed rods in longitudinal direction using electro-discharge machining, as shown in Fig. 1. A displacement rate of 1 mm/min was used for tensile testing, corresponding to an initial strain rate of 3.3 x 10^{-3}/s. The CCD camera as an extensometer was adopted during tensile test.

The micro fatigue testing machine was designed and constructed by authors especially for the miniature specimen. The fatigue testing machine consists of a voice coil motor (BEI Kimco, LA15-16-024A) as actuator, a motor driver (Trust Automation TA115), a load cell with a capacity of 20N (CAS BCL-2L), dynamic amplifier (Vishay, 2310), digital oscilloscope (LeCroy, 9304A), function generator (Agilent, 33220A), a data acquisition system (NI, PXI-8186), and testing machine frame. An analogue output from the DAQ system with LabVIEW software controls the function generator and then the function generator sends the sign signal to the motor drive to the voice coil. One side of the specimen was bent repeatedly by a voice coil actuator, and the other side was connected to a load cell to detect the load magnitude. The detected load signal is amplified by dynamic amplifier and is sent to digital oscilloscope for monitoring the applied load and simultaneously is sent to the DAQ system. The DAQ system receives the signal and calculates the difference between the desired load and the actual load, and changes the voltage to eliminate the error. The frame contains an aligned linear guide for the extension of voice coil, ensuring the colinearity of travel axes, as shown in Fig. 2. The fatigue tests were performed with 15 Hz frequency, in ambient environment at the stress ratio R = -1.

3. RESULTS AND DISCUSSION

Fig. 3a shows the bright field TEM image of unECAPed pure Ti sample. Prior to the ECAP process, the annealed pure Ti (unECAPed) sample contains a low dislocation density. In the TEM images, the low number of straight or crooked dislocations was observed in the large grains of about 150 μm size.

After two pass of ECAP, the slip band width was observed to be thinner than that of the one passed sample, and the band interfaces were distinguished more clearly (Fig. 3b). Meanwhile, the straight-shaped bands varied partially to the crooked shape in the severely strained region. Moreover, the discriminable images with average size of about 0.5 μm were also observed in the intermittent places of the broken slip bands, indicating the most severely strained region. After four pass, recrystallized gains were uniformly distributed throughout the specimen after (Fig. 3c). Phase transformation into the recrystallized grains was considered to be completed

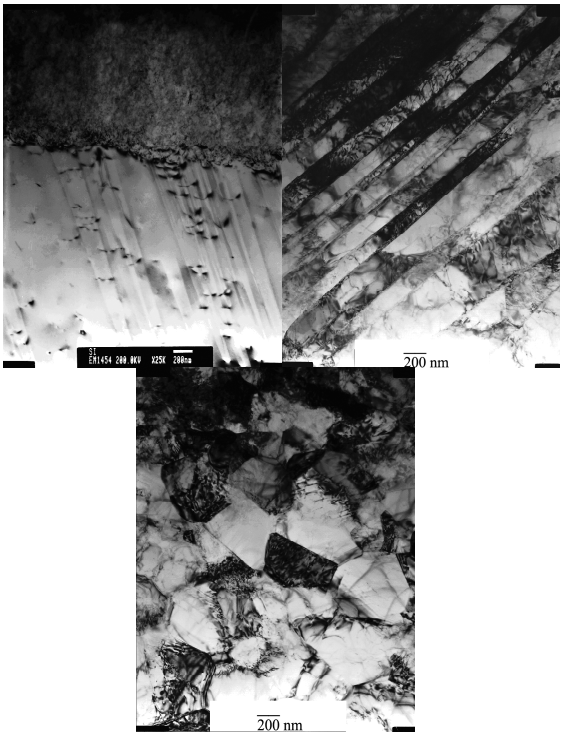

Fig. 3. TEM micrographs of pure Ti (a) unECAPed, (b) 2 pass, and (c) 4 pass ECAP.

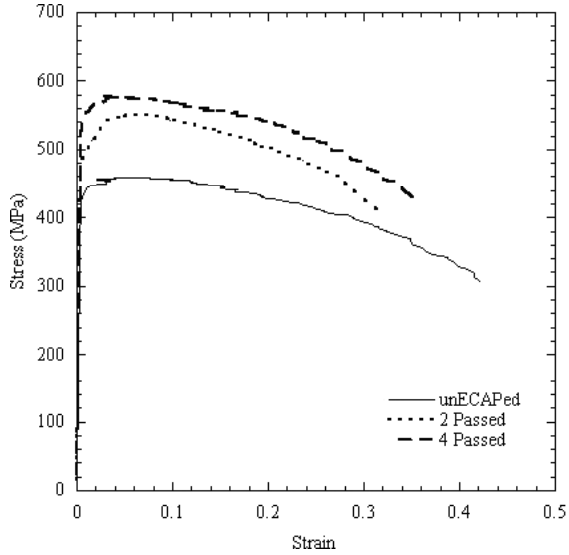

Fig. 4. Tensile stress-strain curves for the unECAPed and ECAPed Ti samples.

throughout the specimen. The average size of the recrystallized grains was approximately 0.4 μm. According to the result by Valiev *et al.* [4] on 8 passed pure Ti with a total strain of 800%, the grain size was 0.3 μm, suggesting that the grain refinement effect with repeated pressing seems to diminish after about 300-400% total strain. Therefore, the grain size reduction following the recrystallization was estimated to be minor by the additional ECAP passes.

The stress-strain curves of the unECAPed, 2 pass and 4 pass ECAPed Ti samples are shown in Fig. 4. Their data for yield stress (YS), ultimate tensile strength (UTS), elongation to failure and grain size are summarized in Table 1. The UTS and YS increase with an increase in the number of pressings. The ultimate tensile strength (UTS) and yield strength (YS) of the 4 pass ECAPed sample were found to be 577 MPa and 513 MPa, respectively. In contrast, the UTS and YS of the unECAPed sample were 459.5 MPa and 388 MPa, respectively. After 4 pass ECAP, the ultimate tensile strength was increased by 26%. This is most likely due to considerable grain refinement through severe deformation by ECAP. This fact suggests that the UFG structure of pure Ti is very advantageous for improving titanium's strength without alloying. The tensile elongation was, however, largely decreased by 14% from unECAPed to 4 passes. This is related to the decrease of strain hardening capability after ECAP, which commonly occurs in other alloys after ECAP [1].

The Hall–Petch relationship correlates the grain size of a material to its yield stress. According the to Hall–Petch relationship, the yield stress σ_y of a material can be expressed as:

$$\sigma_y = \sigma_o + k_y D^{-0.5}, \qquad (2)$$

where σ_o is the lattice friction stress, k_y is the stress intensity for plastic yielding across polycrystalline grain boundaries, and d is the grain size in mm. The values of σ_o and k_y were determined to be 238.3 MPa and 7.5 MPa $\sqrt{\text{mm}}$, respectively, from Fig. 5. The grain size dependence of k_y in the present samples (=7.5 MPa $\sqrt{\text{mm}}$) is much lower than that for pure Ti (=21.2 MPa $\sqrt{\text{mm}}$) [5]. At present it is not clear why there is difference in the value of k_y. This may be partially due to a difference in the impurity content in Ti.

Fig. 6 shows the S–N curves for unECAPed and ECAPed Ti. According to Fig. 6, the fatigue limit σ_e of the pure Ti increased by 29% from 183.1 to 236.1 MPa after 4 pass ECAP. This result indicates that a significant improvement in high-cycle fatigue life can be achieved by applying ECAP on pure Ti. This is in

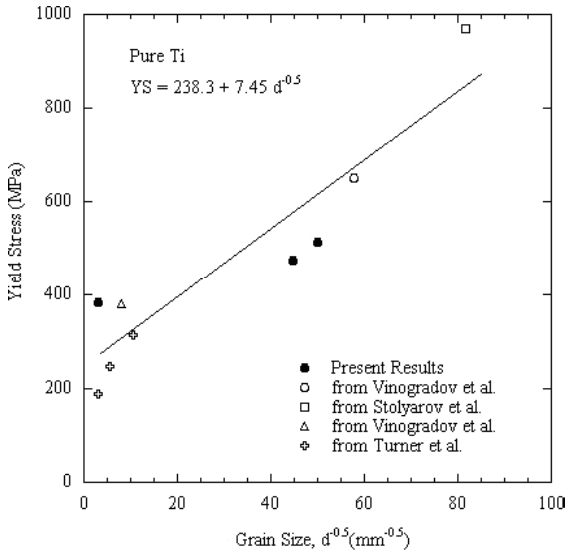

Fig. 5. Hall-Petch behavior of pure Ti.

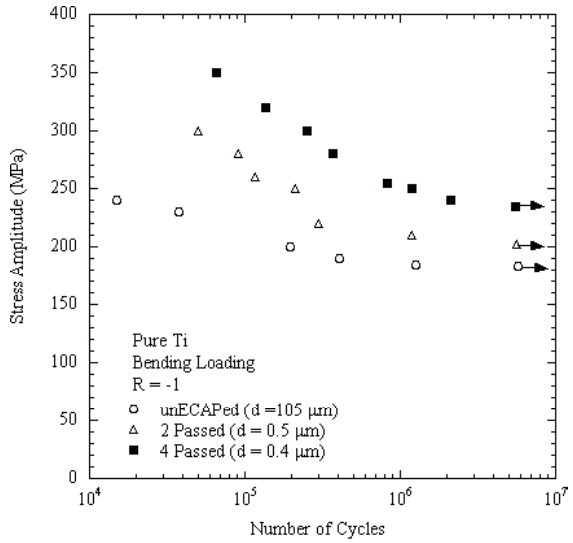

Fig. 6. S–N curves for the unECAPed and ECAPed samples.

contrast to the cases for the Al and Mg alloys after ECAP where little enhancement in high-cycle fatigue performance was observed [4,6]. The tensile and fatigue test results of the unECAPed and ECAPed Ti are summarized in Table 1, together with the data from other investigators for pure Ti [4,7-9].

It is well known that a small grain size will have a positive effect on the yield and tensile strength while generally improving fatigue strength by retarding the crack initiation. The enhanced resistance to crack initiation is considered as a result of high strength of fine grains that act as potential obstacles for dislocation movement, and thereby the formation of microcracks is inactive [10]. The fatigue limits of the current unECAPed and ECAPed Ti are plotted in Fig. 7 as a function of grain size, together with other experimental data for Ti in Table 1 [4,7–9]. As can be seen, the experimental data are reasonably well correlated with $d^{-1/2}$. The values of the fatigue limit, σ_e, in Ti can be described in a similar form to the Hall–Petch relation as:

$$\sigma_e = \sigma_{oe} + k_e d^{-0.5}, \quad (3)$$

where σ_{oe} and k_e are the material constants, and d in mm and σ_e in MPa. The σ_e value continuously increases with decreasing grain size. Present experimental data point lie onto a single straight line depicted by Eq. (3) with σ_{oe} = 78.7 MPa and k_e = 2.5 MPa$\sqrt{\text{mm}}$. The k_e slope of present pure Ti (=2.5 MPa$\sqrt{\text{mm}}$) was one third of k_y of Ti (=7.5 MPa$\sqrt{\text{mm}}$). Similarly, the k_y slope is reported to be larger than that of k_e for copper [11], aluminum [11], alpha brass [11], and low carbon steel [12]. For alpha brass, k_y is nearly twice k_e: k_y = 13.1

Table 1. Mechanical properties and grain size of pure Ti.

Material(Unit)	UTS(MPa)	YS(MPa)	ε_f(%)	σ_e(MPa)	δ(μm)
unECAPed	459.5	383.0	42.1	183.1	105
2 pass	550.5	475	32.3	202.3	0.5
4 pass	5773	513	36.0	236.1	0.4
ECAPed [4]	810	650	15	380	0.3
ECAPed [7]	1050	970	8	420	0.15
unECAPed [8]	460	380	26	238	15
unECAPed [9]	440	315	-	235	9
unECAPed [9]	380	248	-	190	32
unECAPed [9]	377	190	-	178	100

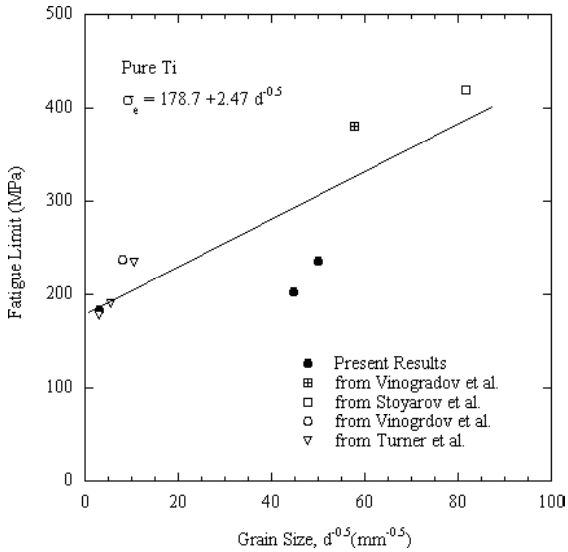

Fig. 7. Hall–Petch behavior of fatigue limit of pure Ti.

MPa $\sqrt{\text{mm}}$ and k_e = 6.9 MPa $\sqrt{\text{mm}}$ [11]. The main factors which affect k_y are dislocation locking and the orientation factor *m*. For titanium, the magnitude of dislocation locking is relatively small. The orientation factor m is related to the number of available slip systems, and because of the limited number of slip system, m can be large in titanium. The larger value of k_y than k_e partially implies that blocking effect of dislocation movement tends to deteriorate in some ways during fatigue loading with grain size refinement, compared to that during static loading. The value of σ_{oe} is reported to be larger than that of σ_{oe} in case of copper [11], aluminum [11], alpha brass [11], and low carbon steel [12]. In contrast, the present Ti exhibits higher σ_0 compared to σ_{oe}. It is not clear why the present Ti exhibits this behavior at present. The influence of grain size on fatigue strength appears to be correlated with ease of cross slip. It is also well known that the fatigue strength of material exhibiting planar slip increases with decreasing grain size [11]. In alpha brass exhibiting planar slip, a difficult cross-slip material, decreasing grain size acts to increase fatigue life. Similarly, grain refinement in pure Ti exhibiting planar slip expects to exhibit considerable enhancement in fatigue limit. In Cu and Al exhibiting wavy slip, in which cross-slip is easy, fatigue life is expected to be insensitive to grain size. However, Cu and Al are reported to exhibit prominent enhancement in their fatigue performance after ECAP [10]. Thus, the effect of grain refinement on fatigue limit in Ti may depend on impurity content and fabrication, in addition to that by slip type.

4. CONCLUSIONS

Yield and fatigue strength dependence on grain size of pure Ti produced by equal channel angular pressing (ECAP) was investigated. Repetitive pressings of the same sample were performed to 4 passes at 683K. After two pass of ECAP, the slip band with a thinner width at average size of about 0.5 mm was observed. After four pass, recrystallized gains were uniformly distributed throughout the specimen with an average size of 0.4 μm. The ultimate tensile strength (UTS) and yield strength (YS) of the 4 pass ECAPed sample were found to be 577 MPa and 513 MPa, respectively. After 4 pass ECAP, the ultimate tensile strength was increased by 26%. The fatigue limit σ_e of the pure Ti increased by 29% from 183.1 to 236.1 MPa after 4 pass ECAP. The standard Hall-Petch relation for yield stress and fatigue limit exhibit in the unECAPed and ECAPed Ti: σ_y = 238.3 MPa + 7.5 MPa $\sqrt{m}\, d^{-0.5}$ and σ_e = 78.7 MPa + 2.5 MPa $\sqrt{m}\, d^{-0.5}$.

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