

# Characterization of microstructure and mechanical property of pure titanium with different Fe addition processed by severe plastic deformation and subsequent annealing

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**Abstract.** Titanium and Ti-alloys are widely used in the marine, aerospace, and biomedical industries due to their high strength to weight ratio, excellent corrosion resistance, and biocompatibility. Increasing the strength level of pure Ti via grain refinement without a considerable decrease in ductility is an attractive approach. Fabrication of nanostructured or ultrafine grained (UFG) metals using severe plastic deformation (SPD) techniques has attracted a lot of interest in the last two decades. The main purpose of the present study is to explore the influence of Fe addition in grain refinement in pure Ti. As-cast pure Ti with two different levels of Fe was firstly deformed by high pressure torsion (HPT) up to 10 rotations at room temperature, and then annealed in a vacuum at a temperature of 500 °C for half an hour. Detailed microstructures were characterized by transmission electron microscopy (TEM) and electron backscatter diffraction (EBSD), and mechanical property was examined by microhardness test.

## 1. Introduction

Severe plastic deformation (SPD) techniques including equal channel angular pressing (ECAP), accumulative roll bonding (ARB) and high pressure torsion (HPT) have drawn significant attention in the last two decades due to their capacity in fabricating bulk metallic materials with nanostructure or ultrafine grained (UFG) structures without changing the dimension of samples [1-3]. Among various SPD techniques, HPT is advantageous for applying very high plastic strains to materials without fracture. During the HPT process, a thin disc sample is placed between two anvils under a high hydrostatic pressure and intense shear strain is introduced by rotating the two anvils with respect to each other. The shear strain  $\gamma$  at a certain radial position in the disc can be calculated as  $\gamma = 2\pi Nr/h$ , where N is the number of HPT rotation, r is the radial distance from the disc center and h is the disc thickness [3].

Ti and Ti-alloys are widely used in the marine, aerospace, and biomedical industries because they have high strength to weight ratio, excellent corrosion resistance and good biocompatibility. Several efforts have been done to fabricate pure Ti with nanostructure or UFG microstructures using HPT process. The first report was conducted by Popov et al. [4] on HPT processing of a commercial pure (CP)Ti (99.53% Ti), and microstructure with an average grain size of about 100 nm was obtained. Then, low temperature annealing of those HPT processed pure Ti was further conducted in other



studies [5,6] to understand its influence on the mechanical properties. Recently, deformation heterogeneity in a Grade-2 CP Ti during HPT process has been extensively investigated in literatures [7-9] in terms of examining the microstructure and microhardness evolutions. Edalati et al. [10] has studied the influence of applied hydrostatic pressure varying from 1.2 to 40 GPa on the microstructural change, microhardness distribution and phase transformation in HPT processed CP Ti. Their results revealed that the phase transformation from  $\alpha$  to  $\omega$  phase occurred when the pure Ti was deformed under pressure larger than 4 GPa and a larger pressure led to a finer microstructure. Edalati et al. [11] has also studied the effect of grain size and HPT processing temperature on the phase transformation in pure Ti based on experimental observations and first-principles calculations. In addition, phase transformation in HPT deformed pure Ti has also been investigated by Todaka et al. [12], considering the influence of impurity oxygen (O). In another study by Todaka et al. [13], effect of the strain path was studied and it has been found that deformations with different strain paths in HPT led to larger Vickers microhardness in pure Ti than monotonic and cyclic HPT processes. Even a number of reports on HPT processing of pure Ti are available, however, influence of impurity in pure Ti on the grain refinement, phase transformation and mechanical properties during HPT process has not been well studied except for the effect of O by Todaka et al. [12]. In the present study, pure Ti with two different levels of impurity Fe, which is the element always included in pure Ti as well as O, is investigated. The pure Ti was firstly deformed by HPT process, and some deformed specimens were further annealed. Microstructure and mechanical property of both deformed and subsequent annealed samples were characterized.

## 2. Materials and experimental details

The experiments in this study were carried out on as-cast pure Ti samples with two different levels of Fe but the same amount of O, namely Ti-0.04%Fe (Ti-0.04O-0.04Fe in mass%) and Ti-0.25%Fe (Ti-0.04O-0.25Fe in mass%). The average colony size of the lamellar  $\alpha$  structures in the as-cast Ti-0.04%Fe and Ti-0.25%Fe was 2.6 mm and 1.5 mm, respectively. Disc samples with a diameter of 10 mm and a thickness of 0.85 mm were cut out from the ingots and provided for the HPT process, which was conducted at room temperature at a speed of 0.5 revolution per minute (rpm) under a compressive pressure of 6 GPa. The disc samples were deformed to 1/4, 1, 5 or 10 HPT rotations (N). The samples processed by 10 HPT rotations (N=10) were selected for further annealing in a vacuum at a temperature of 500 °C for half an hour.

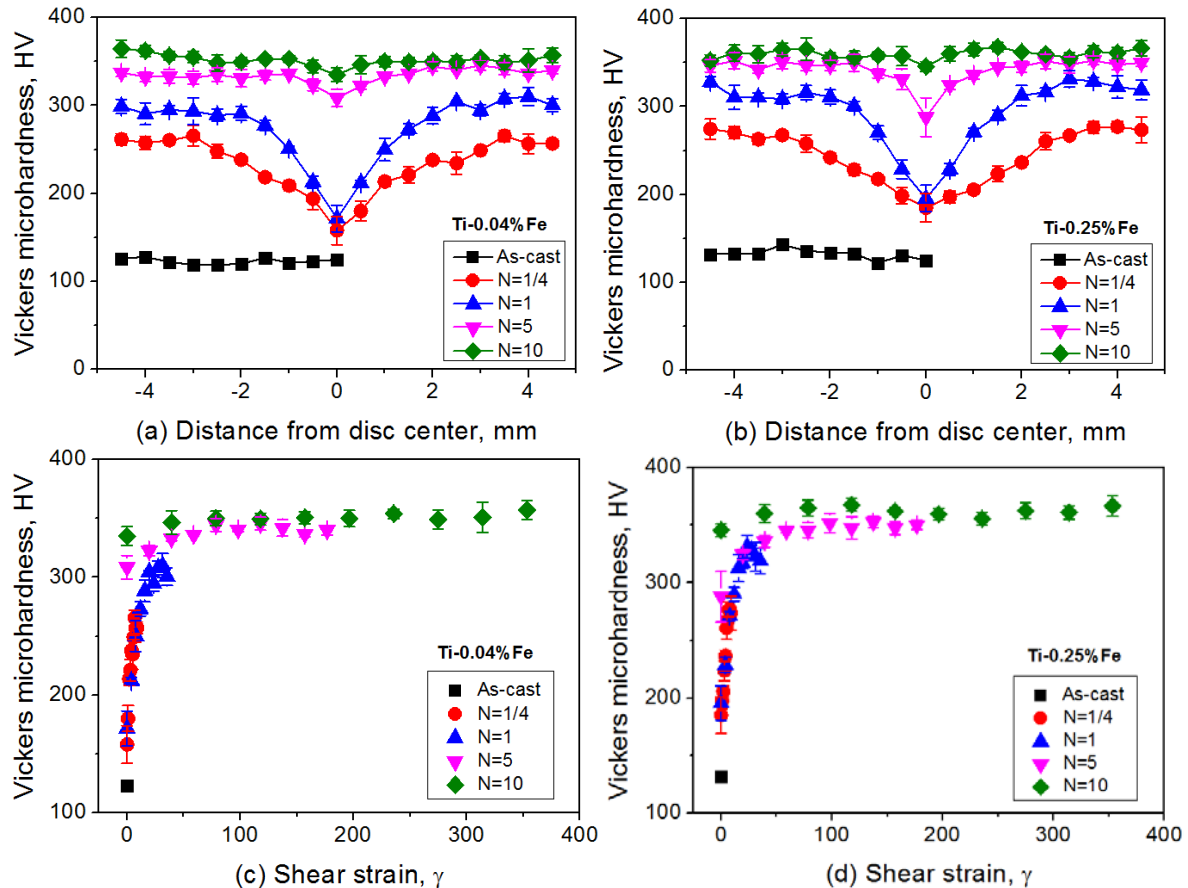
Both the HPT processed and annealed disc samples were firstly polished to obtain mirror-like surfaces. Then the Vickers microhardness was measured with an applied load of 200 g and a dwell time of 15 s across the diameter of each disc on the section perpendicular to the normal direction of the disc. Hereafter, RD, SD and ND indicates the radial direction, shear direction and normal direction of the disc, respectively. Deformed microstructures of the pure Ti specimens were characterized by transmission electron microscopy (TEM) at the position of 3 mm away from the disc center using thin foils parallel to the SD-ND plane, and the annealed microstructures were characterized by electron backscatter diffraction (EBSD) at the position of 3 mm away from the disc center on the RD-SD plane.

## 3. Results and discussion

### 3.1. Microhardness heterogeneity after HPT process

Figure 1 shows Vickers microhardness distributions in the pure Ti specimens deformed by HPT process up to N=10 rotations. As can be seen in Fig. 1a, the average hardness of the as-cast Ti-0.04% is about 123 HV. The HPT deformation leads to a rapid rise of the microhardness. When N is less than 5, the microhardness is lowest at the center of the disc and the hardness increases quickly with increasing the radial distance from the center. The heterogeneity in the microhardness distribution in Ti-0.04%Fe decreases gradually with increasing the number of HPT rotation and relatively uniform microhardness distribution is observed after 5 HPT rotations. With further deformation up to 10 rotations, the microhardness increases slightly to about 355 HV. In Fig. 1b, the microhardness of Ti-

0.25%Fe is about 132 HV before HPT deformation and increases to about 370 HV after 10 HPT rotations. It has been found that the microhardness difference along the diameter after 5 HPT rotations is larger in Ti-0.25%Fe than that in Ti-0.04%Fe. The heterogeneous distribution in hardness remains till 5 rotations and the hardness distribution becomes homogeneous only after 10 rotations in Ti-0.25%Fe.

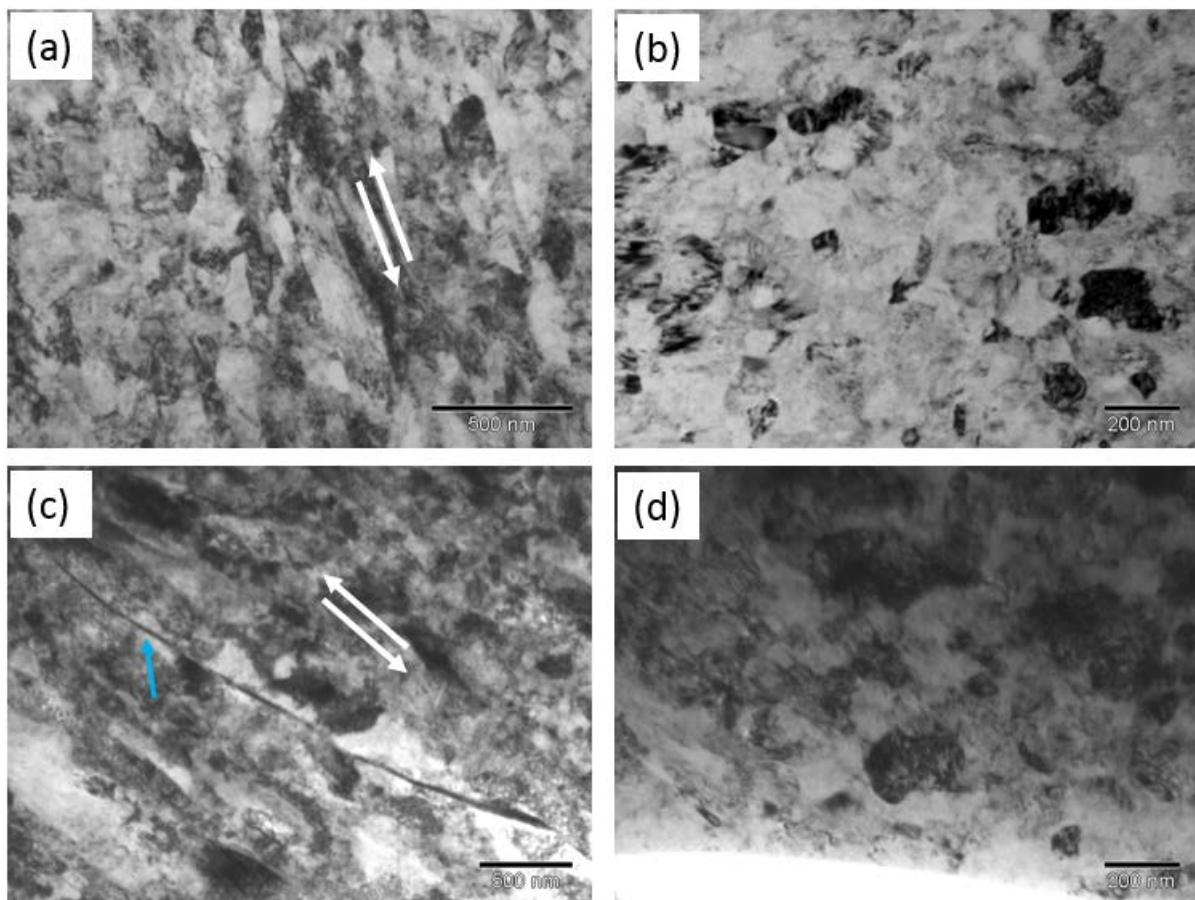


**Fig. 1** Vickers microhardness distributions against radial distance from the disc center in the HPT processed (a) Ti-0.04%Fe and (b) Ti-0.25%Fe, and microhardness plotted as a function of shear strain in the HPT processed (c) Ti-0.04%Fe and (d) Ti-0.25%Fe.

The microhardness evolution in the HPT process has been plotted as a function of shear strain ( $\gamma$ ) in Fig. 1c and d for Ti-0.04%Fe and Ti-0.25%Fe, respectively. It is evident that the microhardness in Ti-0.04%Fe increases significantly with increasing shear strain up to  $\gamma=39.25$ , and then increases slightly above  $\gamma=40$ . On the other hand, the microhardness in Ti-0.25%Fe increases up to  $\gamma=78.5$ , and then saturates. Therefore, it can be concluded that higher Fe contents in pure Ti leads to larger critical shear strain for the microhardness saturation during HPT process. The final hardness values achieved by  $\gamma=353.25$  are 357 HV and 366.5 HV for Ti-0.04%Fe and Ti-0.25%Fe, respectively. In the previous study by Edalati et al. [10], the microhardness in a commercial pure Ti reached to only about 320 HV, which is much lower than the present value of Ti-0.25%Fe even though the amount of Fe in pure Ti and the HPT processing parameters are the same. The different microhardnesses values might be attributed to the different level of impurity O, which is about 0.2% in Ref. [10] but 0.04% in Ti-0.25%Fe in the present study. It has been reported by Todaka et al. [12] that  $\alpha$  to  $\omega$  phase transformation during HPT processing of pure Ti is suppressed by the increase in O content while the extra-hardening of pure Ti is brought by the formation of  $\omega$ .

### 3.2. Deformed microstructures

Figure 2a and b show TEM microstructures of Ti-0.04%Fe HPT processed to N=1/4 and N=10 rotations, respectively. The shear strains at the positions of observations are  $\gamma=5.89$  and 235.5, respectively. After N=1/4 rotation, ultrafine lamellar microstructures elongated along the shear direction indicated by white arrows in Fig. 2a are observed. When the Ti-0.04%Fe sample is further deformed up to 10 HPT rotations, more equiaxed nanostructure with unclear boundaries develops. The average grain size of the equiaxed nano-grains is estimated as 125 nm. Both nanostructures in Fig.2a and 2d involve high density of dislocations.



**Fig. 2** TEM microstructures of Ti-0.04%Fe after (a) N=1/4 and (b) N=10 HPT rotations, and of Ti-0.25%Fe after (c) N=1/4 and (d) N=10 HPT rotations. The microstructures were observed at 3 mm radial position from the center on the SD-ND planes of the HPT discs. The white arrows in (a) and (c) indicate the shear direction in HPT, and the blue arrow in (c) points out the original  $\beta$  phase.

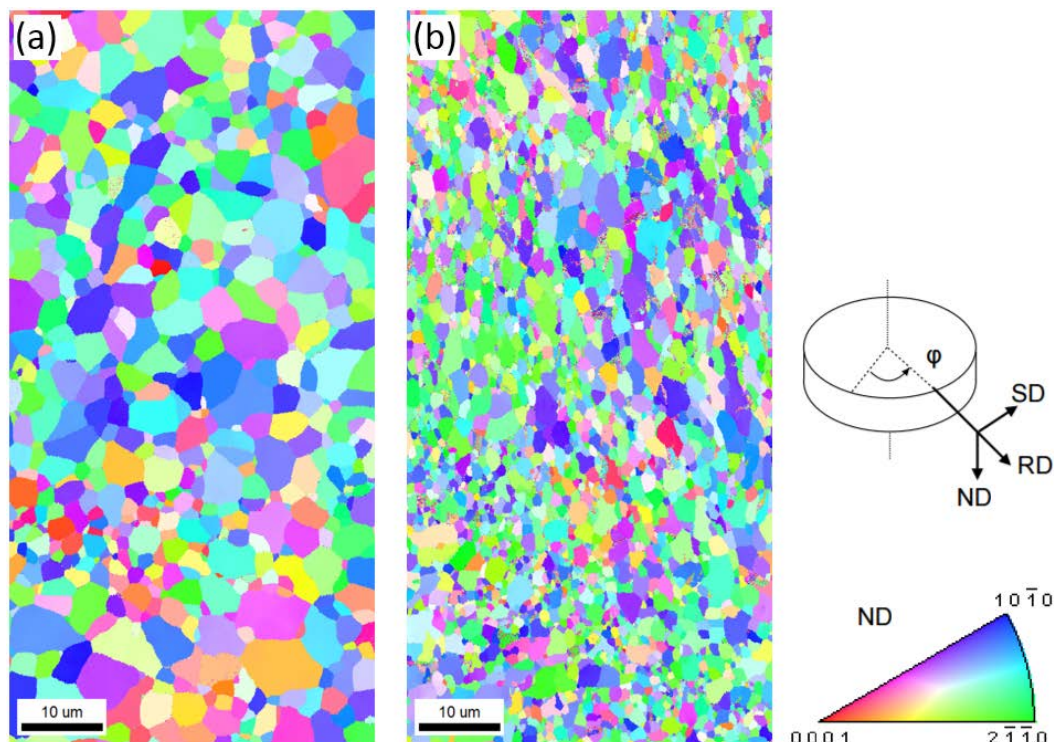
TEM microstructures of Ti-0.25%Fe HPT deformed by N=1/4 and N=10 rotations are shown in Fig.2c and 2d, respectively. Similar microstructure evolution to that in Ti-0.04%Fe is observed. Ultrafine lamellar microstructures develop first (Fig.2c) and they are broken into equiaxed nano-grains when the shear strain becomes very large (Fig.2d). The nanostructure contains large densities of dislocations and the grain boundaries are ill-defined and relatively diffuse. The average grain size of the equiaxed nano-grains in Ti-0.25%Fe is about 100 nm. The microstructures in Fig. 2b and d are similar to the nanostructures shown in early reports [1-13] on HPT processing of commercial pure Ti, where the average grain size varies from ~54 nm to 300 nm depending on the chemical compositions, disc dimension, applied hydrostatic pressure, number of HPT rotations and the rotating speed.



Comparison between Fig. 2b and Fig. 2d indicates that the Fe addition in pure Ti is effective in grain refinement in SPD.

### 3.3. Annealing after HPT process

As observed in Fig. 2, the deformed microstructures in pure Ti after the HPT process contain high densities of dislocations, which are typical nanostructures obtained by SPD. However, such deformed characteristics is one of the main reasons for limited tensile ductilities in SPD processed materials. On the other hand, fully recrystallized nanostructures managing both high strength and good ductility have been reported in some materials [14-16]. For example, Zheng et al. [17] have recently reported fully recrystallized UFG structures exhibiting excellent strength and tensile ductility in a Mg-Zn-Zr-Ca alloy through HPT and subsequent annealing under adequate conditions. In order to obtain fully recrystallized UFG structures in the present materials, the samples after 10 HPT rotations were further annealed at 500 °C for 30 minutes.



**Fig. 3** EBSD orientation maps showing crystallographic orientations parallel to ND of the (a) Ti-0.04%Fe and (b) Ti-0.25%Fe disc specimens HPT deformed by 10 rotations and then annealed at 500 °C for 1.8 ks. Observed at the radial position of 3 mm away from the disc center on the RD-SD plane.

Figure 3a and b are EBSD orientation maps showing annealed microstructures of Ti-0.04%Fe and Ti-0.25%Fe, respectively. It is found that both two samples have fully recrystallized microstructures with equiaxed morphologies after 500 °C annealing. It is evident that the higher Fe content in pure Ti leads to the finer grain size. The average grain size is 3.93 μm in Ti-0.04%Fe and 1.96 μm in Ti-0.25%Fe, respectively.

The Vickers microhardness distribution in the annealed pure Ti discs is quite uniform and the average value is 153 HV in Ti-0.04%Fe and 166 HV in Ti-0.25%Fe, respectively. Significant decrease in the microhardness compared to that in the as-deformed samples is mainly attributed to recrystallization and grain growth as well as phase transformation during subsequent annealing after HPT deformation. It has been reported that ω phase formed in HPT deformation in pure Ti transforms

to  $\alpha$  phase when the annealing temperature is higher than 150 °C [10,12]. However, the influence of Fe addition on the  $\omega$  to  $\alpha$  phase transformation temperature needs to be systematically studied in future.

#### 4. Conclusions

Pure Ti with two different Fe contents were deformed at room temperature by HPT process up to 10 rotations. It was found that the higher Fe content led to the higher Vickers microhardness in the HPT processed discs and also the larger critical shear strain required for the microhardness saturation. Ultrafine lamellar microstructures were observed after 1/4 HPT rotation and equiaxed nano-grains were obtained after 10 HPT rotations in both materials. The average grain size in pure Ti having the larger amount of impurity Fe was smaller at both as-HPT deformed and subsequently annealed (fully recrystallized) states.

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