Characterizations of Temperature-Dependent Tensile Deformation and Fracture Features of Commercially Pure Titanium

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Tensile deformation and fracture behavior of a commercially pure (CP) titanium were investigated at different temperatures through mechanical tests, microstructural observations and fractal analyses. It was found that, with increasing temperature, the number and size of microvoids formed along shear bands (SBs) or at the intersections of SBs on the deformed specimen surface increase, and the fractal dimensions of the scanning profile at the surface near fracture increase correspondingly, and the ones measured perpendicular to the tensile direction is obviously larger than those parallel to the tensile direction, indicating an increased concentration of plastic deformation of CP Ti along the tensile axis. The diameter and depth of dimples on the fracture surfaces of CP Ti increase significantly with increasing temperature, giving rise to a higher fracture surface roughness reflected by a higher fractal dimension. TEM observations demonstrated that the plastic deformation of CP Ti is gradually occupied by dislocation slipping rather than twinning with increasing temperature. This is in good agreement with the fractal analyses of the deformation and fracture features.

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1. Introduction

Titanium and its alloys are widely used in the marine, aerospace, automobile and biomedical industries due to their high strength-to-weight ratio, high melting temperature, corrosion resistance and biocompatibility.1,2 Chen et al.3 reported that commercially pure (CP) titanium has low ductility at room temperature (RT), and requires thermal activation to increase its ductility and formability for lack of operative slip systems, which is characteristic of hexagonal close-packed (HCP) crystals. Similar results were also reported on the tensile deformation of CP Ti at different temperatures.3,4 Tan et al.5 found that many cavitations formed at grain boundaries (GBs) of triple points during tensile tests of a CP Ti alloy at relatively low temperatures (below 873 K (600°C)), but such a phenomenon was not observed at higher temperatures (e.g., 1023 K (750°C) and 1073 K (800°C)), because most defects can be sintered when deformed at temperatures above 1023 K (750°C). Chichili et al.6 and Nemat-nasser et al.7 reported that the deformation twinning is a very important deformation mode during the tensile deformation of pure Ti at different temperatures, and that the density of twins increases markedly with increasing strain rate and decreasing temperature. Recently, the compressive deformation behavior and damage characteristics of ultrafine-grained (UFG) pure Ti and coarse-grained (CG) pure Ti were investigated under different temperatures, and it was found that deformation damage morphologies strongly depend on the testing temperature.8 More recent work by Tsao et al.9 has demonstrated that, as CP Ti sheet was subjected to uniaxial warm tension at temperatures ranging from 623 K (350°C) to 773 K (500°C) and at strain rates from 5.0 × 10⁻² s⁻¹ to 8.3 × 10⁻⁴ s⁻¹, its flow stress decreases with increasing deformation temperature and decreasing strain rate, and the flow deformation behavior exhibits an obvious recrystallization softening characteristic at high temperature and low strain rate.

Despite the fact that there have been many studies on the mechanical deformation behavior of CP Ti, there is little work on systematic qualitative and quantitative characterizations on the temperature-dependent deformation and fracture features of CP Ti. As is well known, fractal geometry has been widely applied to the interpretation of mechanical deformation behavior of materials.10-19 Venkatesh et al.19 established the relationships between the tensile properties and fractal dimension of fracture surfaces of Ti-6Al-4V extra-low interstitial alloy by using scanning electron microscope stereoscopy coupled with three-dimensional surface measurements.18,20 They found that both yield strength (σYS) and ultimate tensile strength (σUTS) decrease, while ductility increases with increasing fractal dimension under different heat treatment conditions. Hilders et al.21 reported that the fractal dimension decreases as the dimple size of the tensile fracture surface decreases for a duplex stainless steel under different heat treatment conditions. The experimental results obtained by Wang et al.22 showed that the ultimate tensile strength of aluminum alloy 6061 composite subjected to tension increases with increasing fractal dimension. These existing research findings strongly demonstrated that the quantitative analysis of deformation and damage features by a fractal method is of particular significance for better understanding of mechanical deformation mechanisms of materials.

In the present work, the tensile deformation and damage behavior and microstructures of a CP titanium were investigated at different temperatures, and quantitative fractal analyses of deformation and damage morphologies were carried out by the yard-stick method23,24 based on Mathemtica programming.

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2. Experimental Procedures

The material adopted is the CP titanium of commercial (99.6% pure) grade, and its average grain size is about 35 µm. The dimension of gauge section of tensile specimens is 5 mm x 5 mm x 25 mm and the total length is 70 mm. Before mechanical tests, all specimens were electro-polished to obtain a strain-free and smooth surface for microscopic observations.

Tensile tests were carried out using a CMT515 testing machine (made in China) at different temperatures (298 K (25°C), 373 K (100°C), 473 K (200°C) and 573 K (300°C)) with a fixed strain rate of 10⁻² s⁻¹. For each high-temperature test, the temperature was gradually raised at a rate of ~10°C/min. After the testing temperature is reached, the sample was first kept at this temperature for 10 min, and then deformed under tension. The temperature was controlled with an accuracy within ±1°C. After mechanical tests, the surface deformation characteristics and fracture surface morphologies were observed by scanning electron microscopy (SEM). The fractal dimensions of scanning profiles D_f on the fracture surfaces and D_s on the lateral surface near fracture of CP titanium at different temperatures were estimated by the yard-stick method based on Mathematica programming. The fractal dimension D_f corresponding to the whole fracture was obtained by averaging over the values for three representative fields of view mutually at 120°. The scanning profiles on the surface close to fracture were obtained almost along or perpendicular to the tensile direction. The extraction of the scanning profile was made by using Digital-Micrograph software, and the obtained profiles were then reconstructed in Mathematica programming. Finally, the fractal dimension D can be evaluated from the relationship between the length of the scanning profile L and the length scale η (the size of the measuring unit), i.e., L = L_0η^{−(D−1)}, where L_0 is the apparent projected length of scanning profile.

Thin foils for transmission electron microscopy (TEM) observation were first sliced from the tensile specimens by spark-machining parallel or perpendicular to the loading direction, then mechanically thinned down to about 80 µm in thickness and finally polished by a conventional twin-jet method at ambient temperature.

3. Experimental Results and Discussion

3.1 Tensile stress–strain response

The tensile true stress–strain curves of CP titanium at different temperatures are presented in Fig. 1. Apparently, the plastic flow behavior of the material under different temperatures is slightly distinctive. The plastic flow curve after yielding appears at room temperature is roughly smooth, whereas a small deflection appears in the early stage of the plastic flow at 373 K (100°C), and such a defective phenomenon becomes more noticeable with increasing temperature (as indicated by the arrows in Fig. 1), implying that as the temperature rises, some new slip systems may be activated or certain twins are possibly formed in some oriented grains.

Clearly, with increasing temperature, the tensile yield strength decreases slightly and the ultimate tensile strength decreases notably, but the elongation increases slightly, as shown in Fig. 2. This is a normal case relating to the easy yielding as a result of enhanced thermal softening with increasing temperature.

3.2 Characterizations of surface deformation damage and fracture surfaces

Figures 3 and 4 show SEM images of the deformation features on the lateral surface near fracture and fracture surface features in CP titanium at different temperatures, respectively, together with the corresponding fractal analyzing results. As a representation, profiles for fractal analyses scanned from the lateral surfaces and fracture surfaces of CP titanium at 473 K (200°C) are clearly presented in Fig. 4.

In general, with increasing temperature, ups and downs on the sample surface become larger and the deformation trace of grains is stretched more seriously along the loading direction (see Fig. 3). Surface deformation damage behavior that occurs with an increase of temperature is described in details as follows. At room temperature, micro-voids appear along the shear direction near fracture (right side (a) of Fig. 3). When the temperature rises to 373 K (100°C), the number of micro-voids formed along the direction of shear bands (SBs) increases significantly. Simultaneously, those voids at the intersections of certain SBs are elongated along the two shear directions, respectively (right side (b) of Fig. 3). As the temperature further increases, the number of
voids continues increasing and the size of voids formed in the SB intersections increases distinctly, and meanwhile, the spacing of grain deformation traces along the loading direction decreases (right side (c) and (d) of Fig. 3), indicating that the material has undergone a severer plastic deformation.

For describing quantitatively such deformation features on the surface, the fractal dimensions of the scanning profile perpendicular and parallel to tensile direction at the surface near fracture, as well as at fracture surface (c) of the CP titanium at 473 K (200°C),

Fig. 4 Representative scanning profiles obtained perpendicular (a) and parallel (b) to the tensile direction, respectively, at the lateral surface near fracture, as well as at fracture surface (c) of the CP titanium at 473 K (200°C).

than those obtained parallel to the tensile direction. Figures 4(a) and 4(b) give the representative scanning profiles obtained perpendicular to and parallel to the tensile direction, respectively, at the surface near fracture. Clearly, the undulation (or roughness) of the scanning profile perpendicular to (rather than parallel to) the tensile axis is larger. Such quantitative measurements above strongly demonstrate that an enhanced concentration of plastic deformation of CP Ti along the tensile axis would take place with increasing temperature, hence leading to an increased tensile elongation (Fig. 2).

Why the fractal dimension increases with increasing temperature can be understood from the following two aspects. On the one hand, the surface roughness results mainly from the different deformation of surface grains. Normally, softer grains with large sizes and preferential orientations will deform and stretch more easily, causing serious depressions locally on the surface. This would become more remarkable with increasing temperature, since the size of some grains may become larger and these grains can thus deform more freely due to the reduced constraints at the surface. On the other hand, the increases in the number and size of voids formed at the intersections of SBs induced by the temperature rising will also cause larger surface roughness.

The fracture surfaces roughness is basically associated with the fracture mode and mechanism, which are strongly dependent upon the mechanical testing conditions. Here, the testing temperature is the critical influencing factor on the tensile fracture surface features of CP Ti. Figure 5 shows SEM images of the fracture surface of CP titanium at different temperatures. It is clear that the tensile fracture surfaces of CP titanium at different temperatures are all composed of dimples; however, with increasing temperature, the diameter and depth of dimples increase. At high temperatures the snake-like slip markings appear in the inner wall of some dimples and even tear ridges form at 573 K (300°C) (Fig. 5), indicating a more serious plastic deformation with increasing temperature. The measured fractal dimensions for fracture surfaces at different temperatures are also presented in Fig. 5. Clearly, with increasing temperature, the fractal dimension $D$ of the scanning profiles on the
Fracture surfaces increase gradually. As one knows, the mode of ductile fracture under high temperature conditions would give rise to a rougher fracture surface, coupled with more energy absorption during the fracture process. Such fractal analyses on the fracture surfaces accounted well for such a phenomenon quantitatively.

From Figs. 2 and 5, it can be found that both yield strength and ultimate tensile strength decrease with increasing fractal dimension. Similar results on 7075-T651 aluminum alloy and Ti-6Al-4V extra-low interstitial alloy were reported by Hilders et al.\textsuperscript{25} and Venkatesh et al.\textsuperscript{18} In a word, the more ductile specimens, corresponding to lower strengths arising from different temperature conditions, were represented by higher values of fractal dimension.

Figure 6 shows TEM images of the tensile deformation microstructures of CP titanium. It can be observed that as temperature increases, the dislocation microstructure evolves from single dislocations or dislocation tangles to dislocation cells, while the number of deformation twins reduces continuously, indicating an enhanced deformation by dislocation slipping and a weakened deformation by twinning. For example, at room temperature, single dislocation loops are observed in CP titanium, and a certain number of deformation twins are clearly observed in local areas (Figs. 6(a), 6(b)). At 373 K (100°C), similar phenomenon as the case of room temperature are observed, but dislocation tangles have now become more obvious (Figs. 5(c), 5(d)). When temperature is raised up to 473 K (200°C), dislocation cells are found in the whole area and the number of deformation twins further decreases (Figs. 6(e), 6(f)). At the highest temperature of 573 K (300°C), more distinct well-developed dislocation cells are found in the whole area and the number of deformation twins further decreases (Figs. 6(g), 6(h)). Similarly, Song and Grey\textsuperscript{26} reported that the lower temperature and higher strain rate promote deformation twinning, and the transition from slipping to twining in the stress–strain behavior of pure Zr is linked to different strain hardening rates. As the temperature increases, the activation energy of slip system decreases gradually, and dislocation motion thus becomes more and more easy and dislocation slip will dominate the entire course of deformation. Also, the results of electron back scatter diffraction (EBSD) observations in uniaxial compression tests of magnesium alloy AZ31B demonstrated that, there are fewer twins in the deformation microstructure at higher temperatures and the deformation mechanism is dominated by slipping rather than twinning with increasing temperature.\textsuperscript{27}

As one knows, the presence of a large number of twins induces the decrease in effective distance of dislocation glide,\textsuperscript{28} and the increase in deformation twinning tendency at low temperatures can be largely attributed to the increase in flow stress,\textsuperscript{29} while the decreased ductility at lower temperatures appear to be connected with the twinning reorientation.\textsuperscript{27} Obviously, the microstructures of the current CP titanium exhibit a temperature-dependent characteristic and the tensile plastic deformation of the material is gradually governed by dislocation slipping rather than twinning with increasing temperature, which is well consistent with the observations on the deformation and fracture features and corresponding fractal analyses.

4. Conclusions

Tensile deformation and damage behavior of CP titanium were investigated at different temperatures and the surface deformation features and fracture surfaces were analyzed by a fractal method. The following concluding remarks can be drawn:

(1) The adopted CP titanium has a high temperature sensitivity of tensile deformation and damage behavior. With increasing temperature, the tensile yield strength and the ultimate tensile strength decreases, but the elongation increases. At room temperature, micro-voids appear along...
the shear direction near fracture. With increasing temperature, the number and size of voids formed at the intersection of SBs increase distinctly. The fractal dimensions of the scanning profile perpendicular and parallel to tensile direction at the surface near fracture were measured to increase gradually with increasing temperature, and the ones measured perpendicular to the tensile direction is obviously larger than those parallel to the tensile direction.

(2) The diameter and depth of dimples on the tensile fracture surfaces of CP Ti increase significantly with increasing temperature, giving rise to a higher fractal dimension, i.e., a higher fracture surface roughness.

(3) The tensile deformation microstructures of CP titanium are strongly dependent upon the testing temperature. With increasing temperature, the dislocation microstructure transforms from single dislocations or dislocation tangles into dislocation cells, and meanwhile, the number of deformation twins reduces continuously, implying that the plastic deformation is gradually occupied by dislocation slipping rather than twinning with increasing temperature. This is in good agreement with the fractal analyses about the deformation and fracture features.

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