



## Full length article

## The effects of ultra-fine-grained structure and cryogenic temperature on adiabatic shear localization in titanium

Zezhou Li <sup>a</sup>, Shiteng Zhao <sup>b</sup>, Bingfeng Wang <sup>c</sup>, Shuang Cui <sup>a, e</sup>, Renkun Chen <sup>a</sup>, Ruslan Z. Valiev <sup>d</sup>, Marc A. Meyers <sup>a,\*</sup>

<sup>a</sup> University of California, San Diego, La Jolla, CA, 92093, USA

<sup>b</sup> University of California, Berkeley, CA, 94720, USA

<sup>c</sup> Central South University, Changsha, 410083, PR China

<sup>d</sup> Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa, 450000, Russia

<sup>e</sup> National Renewable Energy Laboratory, Golden, CO, 80401, USA

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## ABSTRACT

The deformation at low temperatures (173 K and 77 K) in ultrafine-grained (100 and 500 nm) titanium is investigated and its effect on adiabatic shear localization is established. In comparison with coarse-grained titanium, the strength of ultrafine-grained titanium is higher due to the classic Hall-Petch effect while the strain hardening approaches zero. Our results show that shear localization in dynamic deformation is also altered. The width of the shear band of coarse-grained titanium decreases from 30 to 18 μm (by 40%) with decreasing the initial deformation temperature to 77 K. In contrast, for 100 nm titanium, the width of shear band decreases more significantly, from 4 μm at room temperature to 1 μm (a 75% decrease) at 77 K. This difference is attributed to the combined effects of the decrease in the thermal conductivity and specific heat capacity, and the increase in thermal softening rate. These changes in the width are consistent with the predictions of the Grady and Bai-Dodd theories. Ultrafine- and nanocrystallized grains are observed inside the bands which are dependent on initial grain size and initial deformation temperature. The dislocation evolution is evaluated for the different conditions using a Kocks-Mecking formulation; the rotational dynamic recrystallization mechanism responsible for forming the ultrafine/nanosized grains (40–250 nm) is successfully modeled incorporating the differences in initial temperature and grain size. Our results and analysis are important in enhancing the understanding of the structural evolution processes under high strain-rates and cryogenic temperatures.

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## 1. Introduction

Titanium and its alloys have important technological applications such as biomedical implants, airframes, armor, tubing for heat exchangers, and turbines. The dynamic behavior, and especially the microstructure evolution inside adiabatic shear bands formed in coarse-grained titanium and its alloys at room temperature, have been revealed since 1980s [1,2] and studied extensively [3–6]. Over the past 30 years, the production of titanium and its alloys has matured more rapidly than perhaps any other structural material in the history of metallurgy [7,8]. Thus, it is of great significance to examine their mechanical performance in extreme conditions such

as cryogenic temperatures and high strain rates. The mechanical behavior and shear localization mechanisms in coarse-grained (CG) titanium were examined by Meyers et al. [9,10]. Equiaxed ultrafine-grained (UFG) ~200 nm subgrains were observed inside the shear bands which are formed by a rotational dynamic recrystallization mechanism [11,12].

Nanostructured materials have been the subject of widespread research over the past decades [13–16]. The increased volume fraction of the intercrystalline regions (grain boundaries and triple junctions) can significantly alter their physical and mechanical properties [17,18]. It has also been observed that, as the grain size is reduced from microcrystalline to nanocrystalline, the slope of the Hall-Petch equation decreases as was predicted in 1982 by Meyers and Ashworth [19]. A variety of models such as the grain-boundary dislocation source-sink by Swygenhoven et al. [20], the thermal-activated grain boundary shearing by Conrad et al. [21], and the

\* Corresponding author.

E-mail address: [mameyers@eng.ucsd.edu](mailto:mameyers@eng.ucsd.edu) (M.A. Meyers).

grain-boundary rotation and coalescence by Haslam et al. [22] were proposed to explain the deformation mechanisms in nanocrystalline materials.

In addition to the quasi-static mechanical response, the dynamic behavior of UFG and nanocrystalline materials, especially shear localization, has been intensively studied [23,24]. Jia and coauthors [25] studied the mechanical response of ultrafine-grained (UFG) 260 nm titanium under compression and tension; this material was prepared by equal channel angular pressing (8 passes) at 450 °C followed by cold rolling and annealing processes. In contrast to coarse-grained titanium, UFG titanium exhibits a limited strain-hardening ability due to the loss of dislocation accumulation mechanisms. At the same time, the strain-rate sensitivity was reduced due to an increase of strength. These combined effects of decrease of strain-rate sensitivity and strain hardening rate promote the occurrence of adiabatic shear localization in dynamic deformation. In addition, Jia et al. [26] reported that shear localization dominated the plastic deformation for nanocrystalline 80 nm iron under both quasi-static and dynamic compression, as strain hardening almost vanishes. Multiple shear bands nucleated, propagated in the planes with maximum shear stress and broadened their widths with the increase of compressive strain. The dynamic deformation and failure of UFG ~100 nm titanium was studied by Li et al. [27]. The work-hardening of UFG titanium disappears after a rapid strain hardening stage while its strain-rate sensitivity is found to be approximately the same as its coarse-grained (CG) counterpart. The microstructure inside the adiabatic shear band was found to consist of a mixture of elongated and equiaxed nanograins.

Ultrafine-grained (UFG) and nanocrystalline bcc iron, being prepared by powder metallurgy, might have weak interfaces which accelerate softening. The non-existence of strain hardening of nanocrystalline iron can trigger the formation of shear bands at a very low strain; thus, shear bands can be easily formed. In contrast, dislocations can still sustain the plastic deformation in ultrafine-grained titanium. Jia et al. [25] reported that apparent softening does occur under uniaxial compression in the UFG-Ti at strains of 20–24%, followed by catastrophic failure. Thus, it still exhibits very promising structural and biomedical applications with its superior strength and some ductility.

However, the combined influence of initial cryogenic deformation temperature and grain size on mechanical behavior and adiabatic shear localization of titanium is not yet understood. This has an important bearing on the underlying mechanism of microstructural evolution. Thus, this investigation mainly concerns two fundamental questions:

1. How does grain size affect the mechanical behavior of titanium under extreme conditions of high strain-rates and cryogenic temperatures?
2. How does cryogenic condition influence the adiabatic shear localization of CG and UFG titanium?

## 2. Experimental procedures

### 2.1. Mechanical testing

The Split Hopkinson (Kolsky) pressure bar was used to investigate the dynamic mechanical response of CG and UFG titanium. Cylindrical specimens, with both length and diameter of 4 mm, were deformed starting at room (293 K) and low (173 K and 77 K) temperatures. Hat-shaped specimens were used to generate high shear strains in a localized region, thus inducing forced shear localization [28]. The dimensions of hat-shaped specimens [9] used

to induce shear bands were the same for each material. All the cryogenic temperature tests were performed inside an enclosed chamber (containing liquid nitrogen). The temperature inside the chamber was measured by a thermocouple after it was stable.

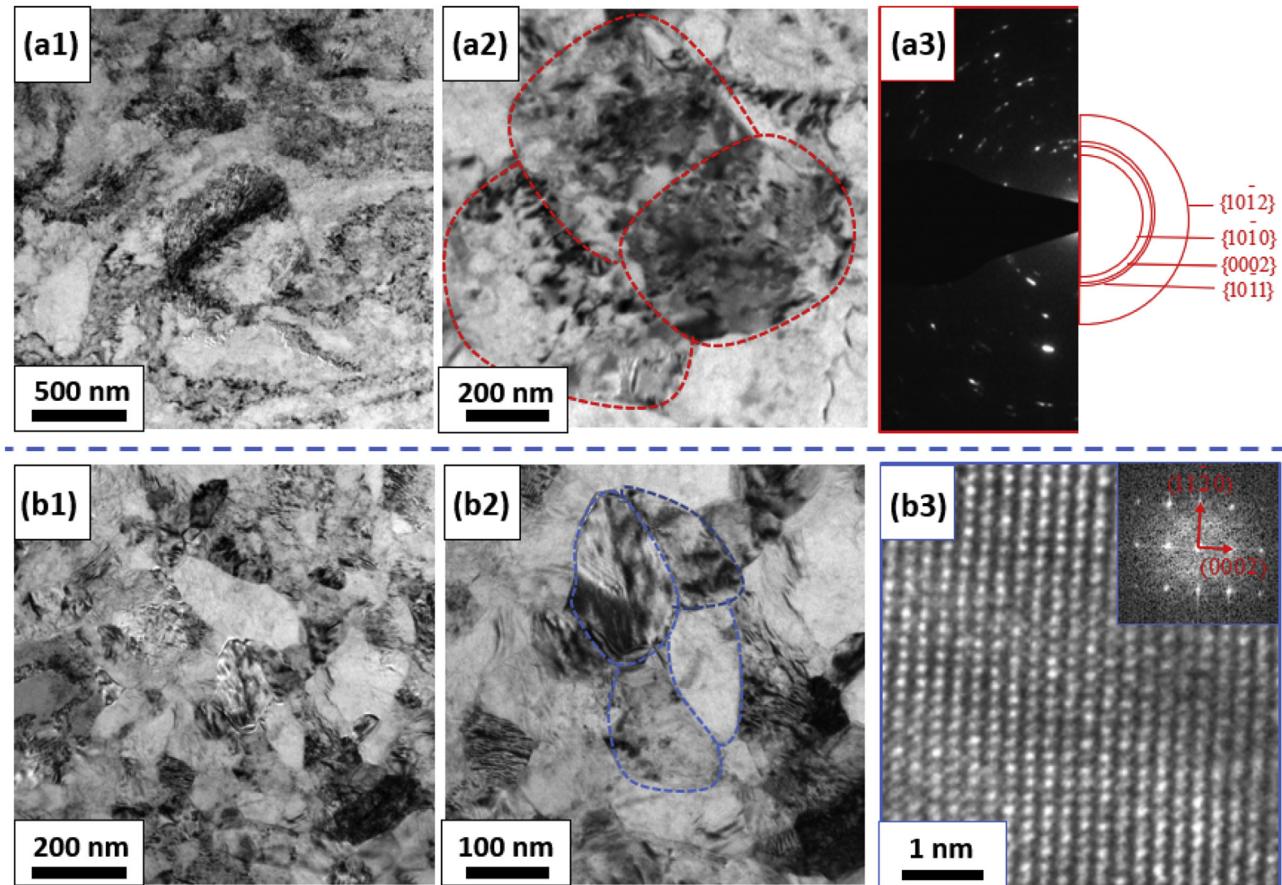
### 2.2. Microstructural characterization

Commercially pure (Grade 2) CG (~10 µm) titanium was chosen for this study, while the as-received UFG titanium rods were prepared by the severe plastic deformation (SPD) technique [7] in two slightly different sequences. The experiments were performed on Grade 4 Ti produced by Dynamet Inc. (Washington, PA, USA) in the form of hot-rolled rods with 15 mm diameter and an average grain size of ~25 µm. The chemical composition of the material, in wt.%, was 0.36% O, 0.15% Fe, 0.05% C, 0.007% N, and 0.0021% H with Ti as the balance. The rods were subjected to processing using the ECAP-Conform facility in Ufa with an angle of 120° in the ECAP abutment. The rod undergoes a total of six passes using route B<sub>C</sub>, when the rods are rotated by 90° in the same sense between each pass, resulting in an ultrafine grain size ~100 nm [29]. In these experiments, the UFG structure was produced by means of ECAP-Conform at 473 K and then drawing at 473 K to a total accumulated strain of ~80%. The deformation process has been described with more details elsewhere [30]. Dyakonov et al. [31] examined the grain refinement mechanism of coarse-grained pure titanium under equal-channel angular pressing, and revealed that mechanical twinning was observed but the contribution of twinning to global grain refinement is relatively limited. Instead, the continuous formation of geometrically-necessary boundaries dominated the microstructural evolution. The post-annealing of severely-deformed UFG titanium was generally able to decrease the dislocation density [32]. Fig. 1(a1) shows the average grain size of the 500 nm UFG titanium. The low dislocation density inside the grains and accumulated dislocations near the grain boundaries in Fig. 1(a1) and 1(a2) indicate that the material underwent dynamic recovery and recrystallization processes during severe plastic deformation. The corresponding selected-area diffraction (SAD) pattern in Fig. 1(a3) shows the polycrystal structure. Fig. 1(b1) shows another as-received UFG titanium with average grain size 100 nm. Fig. 1(b2) shows the equiaxed configuration of grains. The high-resolution image in Fig. 1(b3) confirms its initial HCP crystal structure.

For microstructural examination, the deformed hat-shaped samples were cut parallel to the shear band. The shear bands were examined in a Phillips XL30 scanning electron microscope (SEM). A standard polishing procedure was used to prepare Ø 3 mm transmission electron microscope (TEM) slice samples containing the shear band formed at 293 K for UFG 500 nm titanium. Focused ion beam (FIB) technique was used to accurately extract the TEM sample from inside the narrow shear bands for all the other CG and UFG titanium samples. The FIBed samples were then characterized by JEOL-2800 and Tecnai G<sup>2</sup> Sphera FEI transmission electron microscopes operating at 200 kV.

### 2.3. Thermal conductivity of UFG titanium

The van der Pauw method [33,34] was used to measure the thermal conductivity of four UFG 500 nm titanium samples at room temperature. The electronic component of the thermal conductivity ( $\lambda_e$ ) was extracted from the measured electrical conductivity ( $\sigma_e$ ) via the Wiedemann-Franz law:



**Fig. 1.** (a) Transmission electron microscopy images of Ti with average grain size of 500 nm; (a2) detailed microstructure; (a3) selected area diffraction (SAD) pattern of (a1). (b) Transmission electron microscopy images of Ti with average grain size of 100 nm; (b2) detailed microstructure; (b3) high-resolution TEM image showing hcp crystal structure of Ti.

$$\frac{\lambda_e}{\sigma_e} = L_N T, \quad (1)$$

where  $\lambda_e$  is the electronic thermal conductivity, and  $L_N = 2.44 \times 10^{-8} W \cdot \Omega \cdot K^{-2}$  is the classic Lorenz number for metals based on the Wiedemann-Franz law [35]. Here we use  $\lambda_e$  to represent the total thermal conductivity because it is the dominant contribution in metals at room and low temperatures [36].

### 3. Results and discussion

#### 3.1. Mechanical behavior of UFG titanium

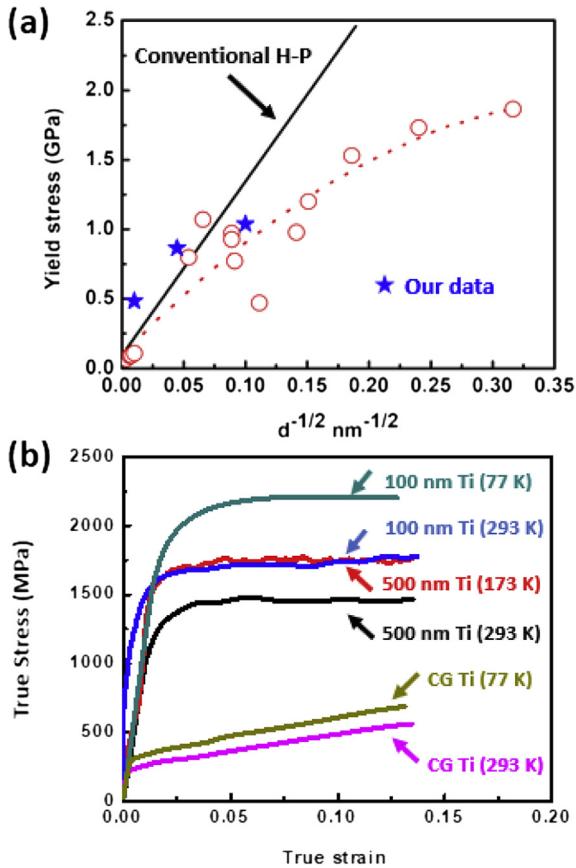
##### 3.1.1. Microhardness

Vickers microhardness tests were performed on the bulk samples, and the values were converted to yield stresses through  $\sigma_y = H_V/3$ . The results in Fig. 2(a) show that the yield stress of UFG titanium increases with decreasing grain size but tends to deviate from the conventional Hall-Petch slope. Sergueeva et al. [37] summarized the microhardness of titanium with different grain sizes and found that it followed the Hall-Petch relationship down to a grain size of 10 nm. However, Meyers et al. [17] showed that the Hall-Petch slope decreases from  $12.5 \text{ GPa nm}^{1/2}$  to  $4.38 \text{ GPa nm}^{1/2}$  in the nano-range. The decrease in the Hall-Petch slope was mainly attributed to different deformation mechanisms gradually becoming dominant. Fig. 2(a) shows that the yield stress for UFG titanium still follows the Hall-Petch relationship [38] but that the slope decreases with decreasing grain size.

##### 3.1.2. Mechanical response of UFG titanium at cryogenic temperatures

Fig. 2(b) shows the true stress - true strain plots of CG and UFG titanium at high strain-rates and various temperatures. It is apparent that the yield stress of both UFG and CG titanium increases with decreasing temperature and increasing strain-rate. In contrast with the clear strain hardening of CG titanium [39], the flow stress of UFG Ti is relatively flat, and no significant strain hardening is observed at either room or low temperatures. Jia et al. [40] reported a transition from strain hardening to strain softening of UFG iron as the grain size changed from  $\sim 1 \mu\text{m}$  to  $\sim 300 \text{ nm}$ ; this apparent strain softening appeared at a very low plastic strain for grain sizes below 300 nm. However, no significant strain softening was observed in UFG titanium under dynamic deformation. In addition, no ductile-to-brittle transition was observed at 77 K in dynamic deformation, indicating that the material can still exhibit significant plasticity.

Ko et al. [41] studied UFG titanium (fabricated by severe plastic deformation process) in tension, and proposed that the absence of strain hardening behavior was caused by a dynamic recovery process balancing the dislocation generation rate with the annihilation rate of trapped lattice dislocations into non-equilibrium grain boundaries. Bengus et al. [42] reported a substantial increase in the tensile yield stress (by a factor of 1.5–2) and decrease in the elongation for UFG ( $\sim 300 \text{ nm}$ ) titanium, when the testing temperature decreased from 300 K to 4.2 K. Nemat-Nasser et al. [43] and Chichili et al. [44] studied the dynamic response of CG titanium over a wide range of temperatures and strain-rates. Nemat-Nasser



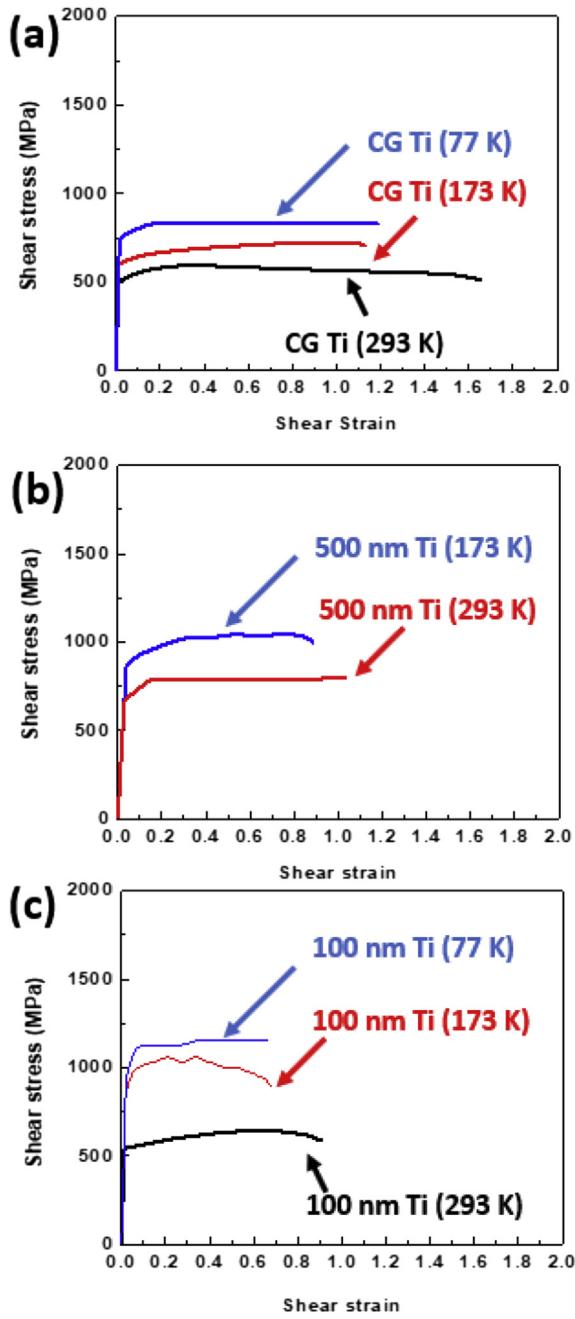
**Fig. 2.** (a) Hall-Petch relationship of titanium from microhardness measurements (blue stars) and comparison with literature results (red circles and line) [17]. (b) Stress-strain curves of Ti with different grain sizes at dynamic loading strain-rates  $\sim 10^3 \text{ s}^{-1}$  [39]. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

et al. [43] reported that the density of twins of CG titanium increased markedly with increasing strain-rate or strain and decreasing temperature. Twin intersections occur and become more pronounced at low temperatures or high strain-rates, which can contribute to the steady work-hardening. In comparison with CG titanium, the strain hardening rate ( $\frac{d\sigma}{d\epsilon}$ ) of UFG titanium was significantly lower. Due to the small grain size of UFG titanium, the capacity for the accumulation of dislocations and twin boundaries significantly decreases. Hence, strain hardening of UFG titanium remains very limited and is far less than that of CG titanium.

The strain-rate sensitivity ( $m = \frac{\partial \log \sigma}{\partial \log \dot{\epsilon}}$ ) of titanium does not change significantly with decreasing temperature, while CG titanium deforms by mechanical twins at 77 K [43] and UFG titanium deforms mainly by dislocation slip. The activation volume for deformation can be expressed as  $V = \frac{kT}{m\sigma_y}$ , where  $k$  is Boltzmann's constant,  $T$  is temperature, and  $\sigma_y$  is the yield stress. Thus, the decrease in temperature and increase in yield stress lead to a decrease in the activation volume of dislocations. This is demonstrated by the rapid plateau of strain hardening in UFG titanium shown in Fig. 2(b).

### 3.2. Adiabatic shear-banding at room and cryogenic temperatures

To examine the grain size and temperature effects on shear localization in titanium, the hat-shaped samples were tested at room temperature (293K) and low temperatures (173 K and 77 K).



**Fig. 3.** (a) Shear stress vs. shear strain relationship in hat-shaped specimens of CG titanium [11]. (b) Shear stress vs. shear strain in hat-shaped specimens of UFG 500 nm titanium. (c) Shear stress vs. shear strain in hat-shaped specimens of UFG 100 nm titanium [27].

The shear stress versus shear strain curves are shown in Fig. 3 [45]. It should be noticed that the shear strain is calculated by the displacement divided by the width of shear region in the hat-shaped specimen. With increasing strain, the flow stress begins to fluctuate due to the competition between strain hardening, strain-rate hardening, and thermal softening. When the thermal softening effect becomes dominant, the stress decreases sharply and a complete adiabatic shear band (ASB) is generated. Fig. 3 indicates that the samples tested at low temperatures were loaded to higher shear stress, in comparison with the samples tested at room temperature, consistently with the mechanical behavior of titanium discussed in Section 3.1.2.

Assuming adiabatic heating, it is possible to estimate the temperature rise within the shear deformation region. Taking into account that a fraction  $\beta$  of the work done is converted into heat, temperature rise  $\Delta T$  is [46]:

$$\Delta T = \frac{\beta}{\rho C_p} \int_{\gamma_s}^{\gamma_e} \tau d\gamma \quad (2)$$

The initial conditions are  $\gamma_s = 0$ ,  $\beta = 0.9$ ,  $C_{p(T=273K)} = 520 \text{ J}/(\text{kg}\cdot\text{K})$ ,  $C_{p(T=173K)} = 430 \text{ J}/(\text{kg}\cdot\text{K})$ ,  $C_{p(T=77K)} = 210 \text{ J}/(\text{kg}\cdot\text{K})$ , and  $\rho = 4500 \text{ kg/m}^3$  [27]. A calculated temperature rise of at least 300 K can be reached for all of the tested samples as determined from Eqn. (2). Since the width of shear band is significantly lower than the shear region of hat-shaped specimens, the actual local shear strain in the shear band is indeed much larger than the values shown in Fig. 3. Using the in-situ infrared detector, at the onset of the load drop, Guo et al. [47] reported that severe strain localization happened first, before the temperature rises to 643–943 K for coarse-grained pure titanium.

### 3.2.1. Width of shear band

The widths of shear bands for CG titanium are shown in Fig. 4(a1, b1, c1). They decrease from ~30  $\mu\text{m}$  at 293 K to ~18  $\mu\text{m}$  at 173 K. In comparison, the shear-band width decreases from ~15  $\mu\text{m}$  at 293 K in Fig. 4(a2) to ~5  $\mu\text{m}$  at 173 K in Fig. 4(c2) for UFG 500 nm titanium. Shear-band bifurcation (within the red dashed lines) was observed at low temperature as shown in Fig. 4(b2). Xue et al. [48] observed this important feature of shear bands in the imploding cylinder geometry; in that case they are geometrically

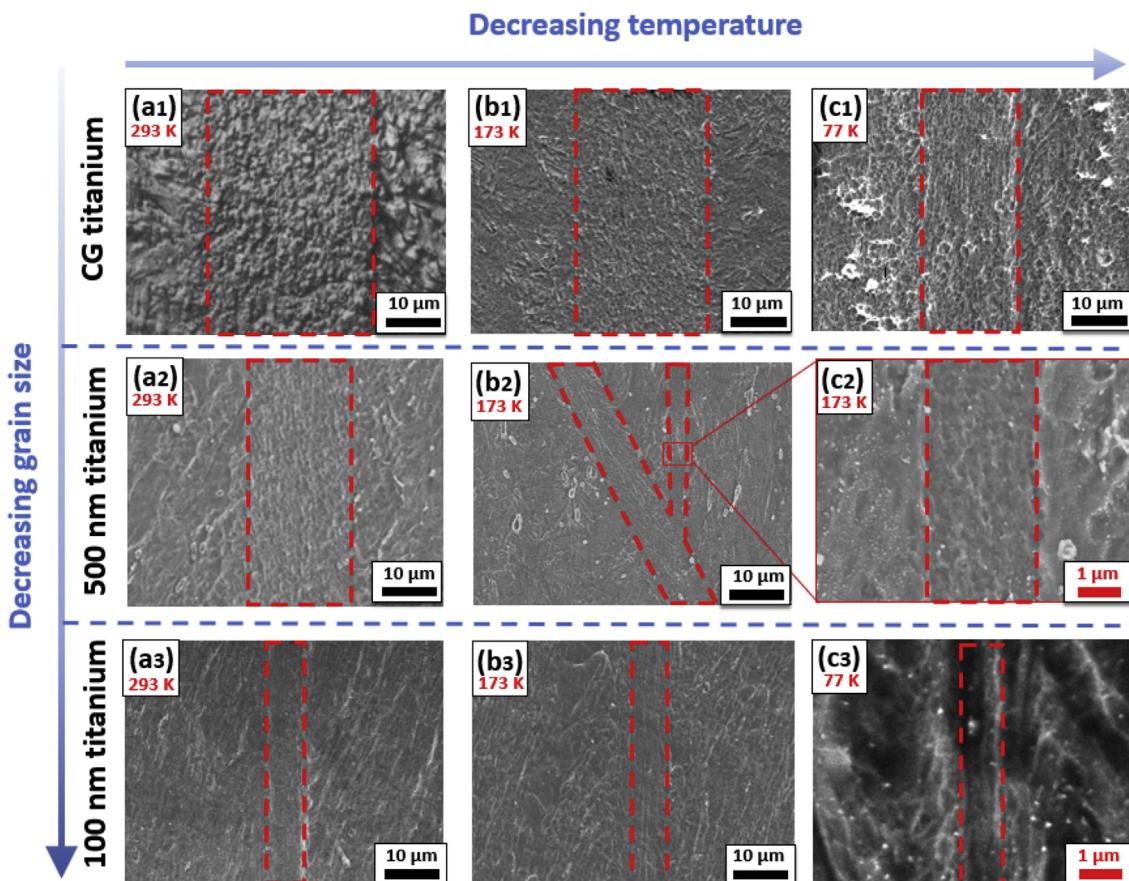
necessary under high strain-rate deformation. For the 500 nm titanium, the width of the shear band is slightly lower at room temperature in comparison with CG titanium. In contrast, the width decreases much more significantly at low temperatures. In addition, there is an obvious drop in the width of shear bands for UFG 100 nm titanium as shown in Fig. 4(a3, b3, c3). They decrease significantly from 4 to 1  $\mu\text{m}$  as shown in Fig. 4(c3). Two theories were proposed to predict the shear-band width; they are used here to evaluate the effects of temperature and grain size. The Bai-Dodd equation [49] predicts the width,  $\delta$ , of the shear bands as:

$$\delta = 2 \left( \frac{\lambda T}{\tau \dot{\gamma}} \right)^{1/2} \quad (3)$$

where  $\lambda$  is the thermal conductivity;  $T$ ,  $\dot{\gamma}$ , and  $\tau$  are the temperature, shear strain-rate and shear stress inside the shear band. Alternatively, Grady [50,51] found that the shear-band width at minimum dissipation corresponded to a maximum shear-band growth rate. He derived the following expression for the width of shear band, incorporating the thermal softening parameter  $\alpha = \frac{1}{\sigma_y} \frac{\partial \sigma}{\partial T}$  [50,51]:

$$\delta = \left( \frac{9\lambda^3}{\tau^3 C_p \alpha^2 \dot{\gamma}} \right)^{1/4} \quad (4)$$

where  $C_p$  is the specific heat capacity [9,27] and the other symbols are defined above. These two classic models predict a reduced width  $\delta$  with increasing strength, strain-rate, thermal softening, specific heat capacity, and decreasing thermal conductivity. The



**Fig. 4.** SEM images showing the width of shear bands for different titanium conditions at different initial deformation temperatures.

sub-sections below show how these parameters vary for the materials under study. They are affected by grain size and temperature.

**3.2.1.1. Effect of thermal conductivity.** Thermal conduction in solids includes electron and phonon contributions. In general, in pure metals, the main contribution to thermal conductivity comes from electrons [36]. The measured electrical resistivity of the thick UFG 500 nm titanium disks was  $0.59 \pm 0.01 \mu\Omega$ . Thus, applying the

Wiedemann-Franz law represented by Eqn. (1), the electronic thermal conductivity is reduced from  $22 \text{ W m}^{-1} \text{ K}^{-1}$  [9] for coarse-grained to  $12.12 \text{ W m}^{-1} \text{ K}^{-1}$  (~45% reduction) for UFG Ti as shown in Fig. 5(a).

Guo et al. [52] examined the thermal conductivity of nanostructured titanium by scanning thermal microscopy that allowed it to be mapped down to the submicron scale. The analysis showed that the thermal conductivity decreased to  $5.2 \text{ W m}^{-1} \text{ K}^{-1}$  for nanostructured titanium from  $22 \text{ W m}^{-1} \text{ K}^{-1}$  for coarse-grained Ti. The thermal conductivity is related to the effective conduction electron density. The volume fraction of triple junctions and grain boundaries is still quite small for ultrafine-grained materials. However, the number of grain boundary barriers per volume is significantly increased with the reduction of grain size down to 100 nm. This leads to a decrease of the effective electron density, leading to the significant decrease of thermal conductivity in ultrafine-grained titanium.

**3.2.1.2. Effect of heat capacity.** Heat capacity of metals is contributed by lattice vibration. Debye [53] assumed that the range of frequencies of vibration available to the oscillators was the same as that available to the elastic vibrations in a continuous solid. The lower limit of the wavelengths of these vibrations was determined by the interatomic distances in the solid. He obtained the following expression for the heat capacity of solids under constant volume:

$$C_v = 9R \left( \frac{T}{\theta_D} \right)^3 \int_0^{\theta_D/T} \frac{x^4 e^{-x}}{(1 - e^{-x})^2} dx \quad (5)$$

where  $x = h\nu/kT$  ( $\nu$  is the Planck's constant),  $R = 8.314 \text{ J/(K \cdot mol)}$  and the Debye frequency is  $\nu_D = 8.75 \times 10^{12} \text{ s}^{-1}$ .  $\theta_D = h\nu_D/k = 420 \text{ K}$  is the characteristic Debye temperature for titanium [54]. Fig. 5(b) shows that the specific heat capacity for both CG and UFG titanium decreases with decreasing temperature due to the lower population of thermally excited phonon modes [55]. Thus,  $C_{T=293K} = 520 \text{ J/(kg \cdot K)}$  decreases to a much lower value of  $C_{T=77K} = 210 \text{ J/(kg \cdot K)}$  at 77 K. The heat capacity does not change significantly when decreasing the grain size down to 100 nm, since the major contribution was due to the lattice vibration. Thus, the relatively small volume fraction of grain boundaries plays a very minor influence. The decrease of specific heat capacity will cause a more rapid increase of temperature for the same deformation energy and contribute to a narrower shear region.

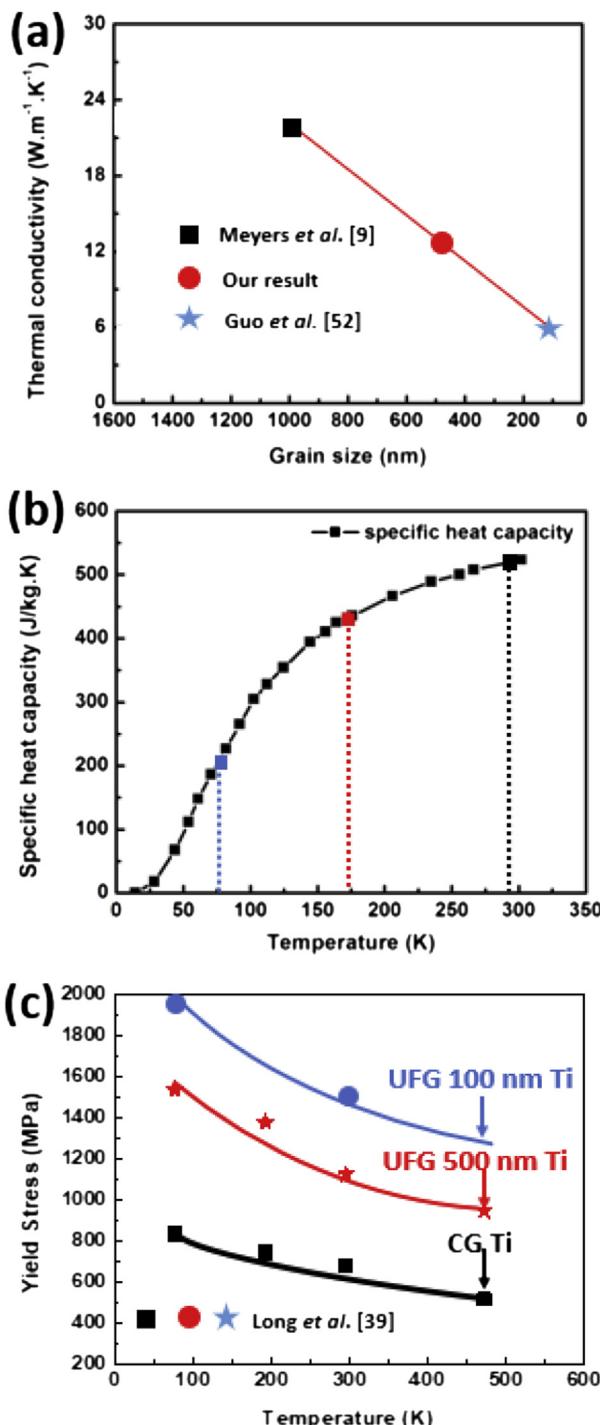
**3.2.1.3. Effect of thermal softening.** The thermal softening parameter:

$$\alpha = -\frac{1}{\sigma_y} \left( \frac{\partial \sigma}{\partial T} \right)_{\gamma, \dot{\gamma}} \quad (6)$$

is obtained from the temperature dependence of the yield stress. To a first approximation, the strain softening of UFG 100 nm titanium ( $\alpha_{UFG} \approx 0.002$ ) is larger than that of CG Ti ( $\alpha_{CG} \approx 0.001$ ) at 77 K [56].

### 3.2.2. Combined effects on shear-band width

Here we discuss the three effects and apply them to the Bai-Dodd and Grady equations. First, the reduction of the thermal conductivity of nanostructured titanium at room temperature, as shown in Fig. 5(a), leads to the decrease in the width of shear band according to Eqn. (3) [49]. It was also reported that the thermal conductivity of titanium does not change significantly when decreasing the temperature to 77 K [57,58]. The lower specific heat capacity at cryogenic temperature transfers more heat under the



**Fig. 5.** (a) Effect of grain size on the thermal conductivity of titanium [9,52]. (b) Theoretical values of specific heat capacity of titanium as a function of temperature. (c) Thermal softening in CG and UFG titanium [39].

same input of plastic work, and low thermal conductivity provides the material a slow diffusion rate of heat to the surroundings. In addition, the increase of the thermal softening parameter contributes to the narrower width of shear band according to Eqn. (4). Also, increased thermal softening and decreased strain hardening can make UFG material more prone to shear localization, which has also been reported in UFG iron [40]. Thus, the higher thermal softening of UFG titanium at 77 K, as shown in Fig. 5(c), also contributes to the decrease in the width of shear bands.

The effects of these parameters are incorporated into the Bai-Dodd and Grady equations and plotted in Fig. 6. The decrease in the width of shear band of UFG titanium at initial cryogenic temperatures can be attributed to the decrease of thermal conductivity, the decrease of heat capacity, and the increase of thermal softening as presented in Fig. 5. Parameters in Table 1 were applied to the Bai-Dodd and Grady equations to predict width of shear bands in both CG and UFG (100 nm) titanium. The predictions of the two equations are provided in Fig. 6, together with the experimental results. The predictions of the Bai-Dodd and Grady theories use the heat capacity at room temperature. The experimental results show a more drastic dependence on strength than the predictions from the Bai-Dodd and Grady equations; for lower strength, they are close to the CG Bai-Dodd equation and for higher strength, they approach the Grady equation. The effects of a decreased thermal conductivity and increased thermal-softening are confirmed.

Hines and Vecchio [59,60] studied shear localization of shock-hardened copper [61] at initial deformation temperatures of 293 K and 77 K and similar levels of deformation. The width of shear bands did not change significantly and was in the range 200–250  $\mu\text{m}$ . However, copper is FCC and exhibits a very low

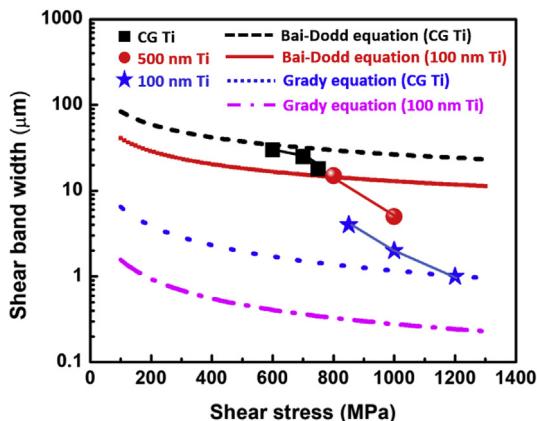
thermal softening component. Thus, the strengths at 293 and 77 K are not significantly different. In the current work, the width of the fully developed shear band of CG titanium dropped from 30 to  $\sim 18 \mu\text{m}$  when the initial deformation temperature was cryogenic, a 40% decrease. In contrast, the width decreases more significantly for both UFG 500 nm and 100 nm titanium at low temperature, from 4 to 1  $\mu\text{m}$ , a 75% decrease. Since the temperature difference inside and outside the shear band does not have too much significance, the heat diffusion-rate effect on width of shear band is not thought to be critical. However, in Eqn. (3), the width of shear band decreases with the decrease of the thermal conductivity. In addition, from Eqn. (4), the width of shear band decreases with the increase of thermal softening parameter. One would expect that an extremely narrow shear band should form with further decreasing temperatures and increasing strain-rates [62–67].

### 3.3. Microstructural evolution inside the shear bands

Fig. 7 shows TEM micrographs from the shear bands. The corresponding selected area electron diffraction (SAED) patterns for each image demonstrate the preponderance of randomly distributed recrystallized grains. The selected area electron diffraction (SAED) patterns do not suggest strong texture, with an apparent absence of concentrated intensity in the ring pattern. The shear bands contain both elongated and equiaxed grains in Fig. 7(a1), which are much smaller than the grains in the matrix. Due to dynamic recrystallization, the overall dislocation density decreases significantly inside the shear bands. Dislocations are again generated because of the continuing deformation (post recrystallization). The average grain size inside the shear band of CG titanium at 77 K is 140 nm as observed in Fig. 7(c1); this is much smaller than the average grain size of 250 nm inside the shear band formed with initial deformation at room temperature of CG titanium [9] (Fig. 7(a1)).

For UFG titanium, the average recrystallized grain size within the shear bands decreases with the lowering of the initial temperature. Fig. 7(a2) shows elongated grains breaking up into recrystallized subgrains. A higher magnification image in Fig. 7(b2) illustrates the formation of equiaxed grains. Fig. 7(c2) shows that UFG subgrains with average grain size of 120 nm were formed inside the shear band with deformation initiated at 173 K. Fig. 7(a3) shows the formation of nano-bundles during the shear deformation in UFG 100 nm titanium at 293 K. The red arrow shows a series of nano-bundles ranging from 30 nm to 80 nm. Fig. 7(b3,c3) illustrate the formation of extremely small nanograins inside the shear bands at cryogenic temperatures. No deformation twins are found inside any of shear bands, further evidence of dynamic recrystallization.

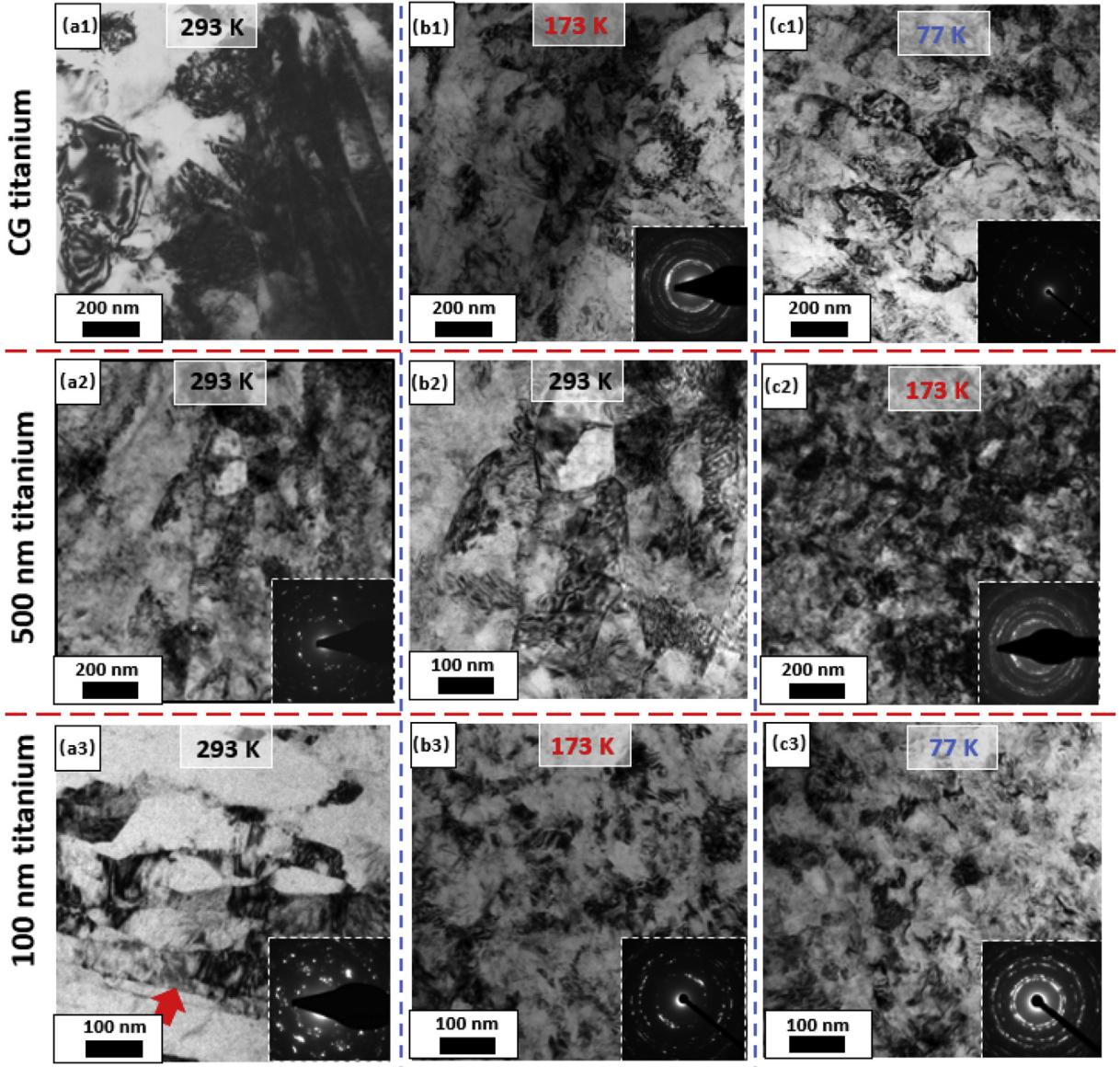
Fig. 8 shows higher magnification images of subgrains inside the shear bands. Very fine recrystallized grains are the dominant feature. Fig. 8(a1) shows that the UFG elongated dislocation cells were formed in the shear band at room temperature for CG titanium. The dark-field image of Fig. 8(b1) shows subgrains with grain size around 140 nm formed in the shear band at 77 K. The dislocations inside the grain are evidence of on-going deformation. Fig. 8(a2) shows the break-up of elongated grains in the shear band of UFG 500 nm titanium at 293 K. Recrystallized grain boundaries and triple points are observed in Fig. 8(b2) in the shear band at 173 K; the grains have narrow and faceted boundaries, which are characteristic of recrystallization [61]. Fig. 8(a3) shows the formation of both nano-bundles and recrystallized equiaxed grains in the shear band of UFG 100 nm titanium with initial deformation at 293 K. The dark-field image of the shear band in Fig. 8(b3) demonstrates the formation of nanosized grains inside the shear band at 77 K.



**Fig. 6.** Width of the shear band in pure titanium for different grain sizes and temperatures: comparison of Bai-Dodd and Grady theories with experimental results [9,49–51]. The full line represents the Bai-Dodd equation prediction applied to 100 nm titanium. The black dotted line corresponds to Bai-Dodd equation for CG Ti. Dash dotted (100 nm Ti) and dotted (GG Ti) lines correspond to Grady theory.

**Table 1**  
Parameters for the Bai-Dodd [49] and Grady [50,51] equations.

Parameters	Values
$\lambda(\text{CG} - \text{Ti})$	$22 \text{ W m}^{-1} \text{ K}^{-1}$
$\lambda(100\text{nm} - \text{Ti})$	$5.2 \text{ W m}^{-1} \text{ K}^{-1}$
$\rho$	$4500 \text{ kg/m}^3$
$C_p(T=293\text{K})$	$520 \text{ J/(kg}\cdot\text{K)}$
$\dot{\gamma}$	$10^5 \text{ s}^{-1}$
$\alpha_{100\text{nm}-\text{Ti}}$	0.002
$\alpha_{\text{CG}-\text{Ti}}$	0.001
T	800 K



**Fig. 7.** TEM images of the shear bands formed in CG titanium at (a1) 293 K [9], (b1) 173 K, (c1) 77 K. TEM images of the shear band formed in UFG 500 nm titanium at (a2) 293 K, (b2) 293 K, (c2) 173 K. TEM images of the shear band formed in UFG 100 nm titanium at (a3) 293 K [27], (b3) 173 K, (c3) 77 K.

The grain size distribution inside the shear bands for the different materials and initial deformation temperatures is depicted in Fig. 9. There are clearly two trends: (1) as the initial grain size is decreased, so it is the recrystallized grain size in the shear band; (2) as the initial deformation temperature is decreased, so is the grain size in the shear band.

#### 3.4. Dislocation-based modeling of microstructure evolution inside shear bands

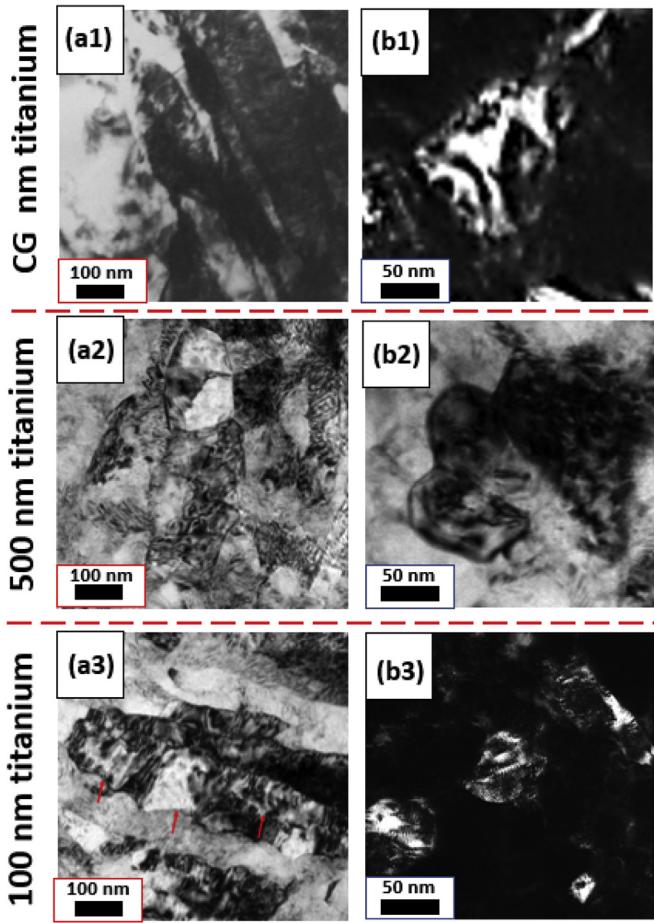
A model that is realistic has to predict the effects of initial deformation temperature and grain size on the nano/microstructure within the shear band. The observations in the previous sections are summarized in Fig. 10. The width of shear band decreases with initial grain size as shown in Fig. 10(a). The recrystallized grain size decreases with the width of shear band as shown in Fig. 10(b).

The local shear strain  $\gamma_{local}$  inside the shear bands was estimated as follows:

$$d_{total} = d_{uniform}\gamma_{uniform} + \delta_{local}\gamma_{local} \quad (7)$$

where  $d_{total}$  is the displacement in specimen,  $\delta_{local}$  is the width of the shear band, and  $\gamma$  is the shear strain. The calculated local shear strains for different shear bands are plotted against the recrystallized grain size in Fig. 10(c). Under the same displacement of hat-shaped specimens, the decrease in the width of the shear band (at cryogenic temperatures) leads to a much larger local shear strain inside the shear bands, which in turn is responsible for the formation of a smaller recrystallized grain size. These are first-order estimates and the local shear strain may be smaller than the calculated ones. However, it is clear that the recrystallized grain size decreases with increasing shear strain inside the band.

The existing rotational recrystallization theory [10,11] does not have this level of detail and therefore needs to be amplified. The evolution of dislocation density with plastic deformation is the result of the combined effects of dislocation generation and annihilation [68]:



**Fig. 8.** Microstructure inside the shear bands for CG Ti at (a1) 293 K [9] and (b1) 77 K. Microstructure inside the shear bands for UFG 500 nm Ti at (a2) 293 K and (b2) 173 K. (a3) Microstructure inside the shear bands for UFG 100 nm Ti at 293 K. The red arrows show the formation of recrystallized subgrains in the elongated nano-bundle. (b3) Dark-field image shows the formation of equiaxed subgrains in shear band at 77 K. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

$$\dot{\rho} = \dot{\rho}_{gen} - \dot{\rho}_{ann} \quad (8)$$

where  $\dot{\rho}_{gen}$  and  $\dot{\rho}_{ann}$  represent dislocation generation and annihilation rates, respectively.

Kocks and Mecking [69] proposed that the evolution of the dislocation density during plastic deformation is due to the competition between cutting forest dislocations and dynamic recovery of dislocations, and obtained the following expression:

$$\frac{d\rho}{d\gamma} = k_h \sqrt{\rho} - f_s \rho \quad (9)$$

where  $k_h$  is a hardening factor and  $f_s$  is a softening parameter. The cutting of forest dislocations leads to strain hardening and the annihilation of dislocations by dynamic recovery results in strain softening. Thus, it is possible to estimate the dislocation density at the recrystallization temperature ( $\sim 0.4 T_m$ ), often considered as required for shear localization.

The energy of randomly distributed dislocations is [10,11]:

$$E_1 = \rho_d \left( \frac{\pi G b^2}{4\pi} \right) \ln \left( \frac{\alpha_0}{2b\rho_d^{1/2}} \right) \quad (10)$$

where  $\pi$  is the constant depending on the character of the dislocation,  $G$  is the shear modulus,  $b$  is Burgers vector,  $\alpha_0$  is a constant which accounts the core energy of the dislocation, and  $\rho$  is the dislocation density, which can be estimated throughout the plastic deformation process from the Kocks-Mecking Eqn. (9).

The energy of low-angle grain boundary arrays with the same dislocation density, arranged in ellipsoidal subgrains is:

$$E_2 = \rho_d \left( \frac{\pi G b^2}{4\pi} \right) \ln \left[ \frac{e\alpha}{4\pi b} \left( \frac{S}{V} \right) \frac{1}{\rho_d} \right] \quad (11)$$

where  $S$  and  $V$  are surface area and volume of ellipsoid. The ellipsoid has a width  $W$  with aspect ratio  $k$ . The surface area  $S$  and volume  $V$  in equation (11) are given by the standard equations [70]:

$$S = 2\pi f(k)(W/2)^2 \quad (12)$$

$$V = \frac{4}{3}\pi k(W/2)^3, \quad (13)$$

$$f(k) = 1 + \frac{k^2}{\sqrt{k^2 - 1}} \sin^{-1} \left( \frac{\sqrt{k^2 - 1}}{k} \right) \quad (14)$$

By setting  $E_1 = E_2$  in Eqns. (10) and (11), one can obtain the critical dislocation density for dynamic recrystallization:

$$\rho_d^* = \left[ \frac{3e}{4\pi} \left( \frac{f(k)}{k} \right) \left( \frac{1}{W/2} \right) \right]^2 \quad (15)$$

It is assumed that when  $k \rightarrow +\infty$  and  $f(k)/k = \pi/2$ , the subgrains are the longest and  $E_1$  reaches a maximum. This leads to the following expression:

$$\rho_d^* = \left[ \frac{3e}{4} \left( \frac{1}{W} \right) \right]^2 \quad (16)$$

Thus, one can calculate the width of the ellipsoidal subgrains,  $W$ :

$$W = \frac{3ep^{-1/2}}{4} \quad (17)$$

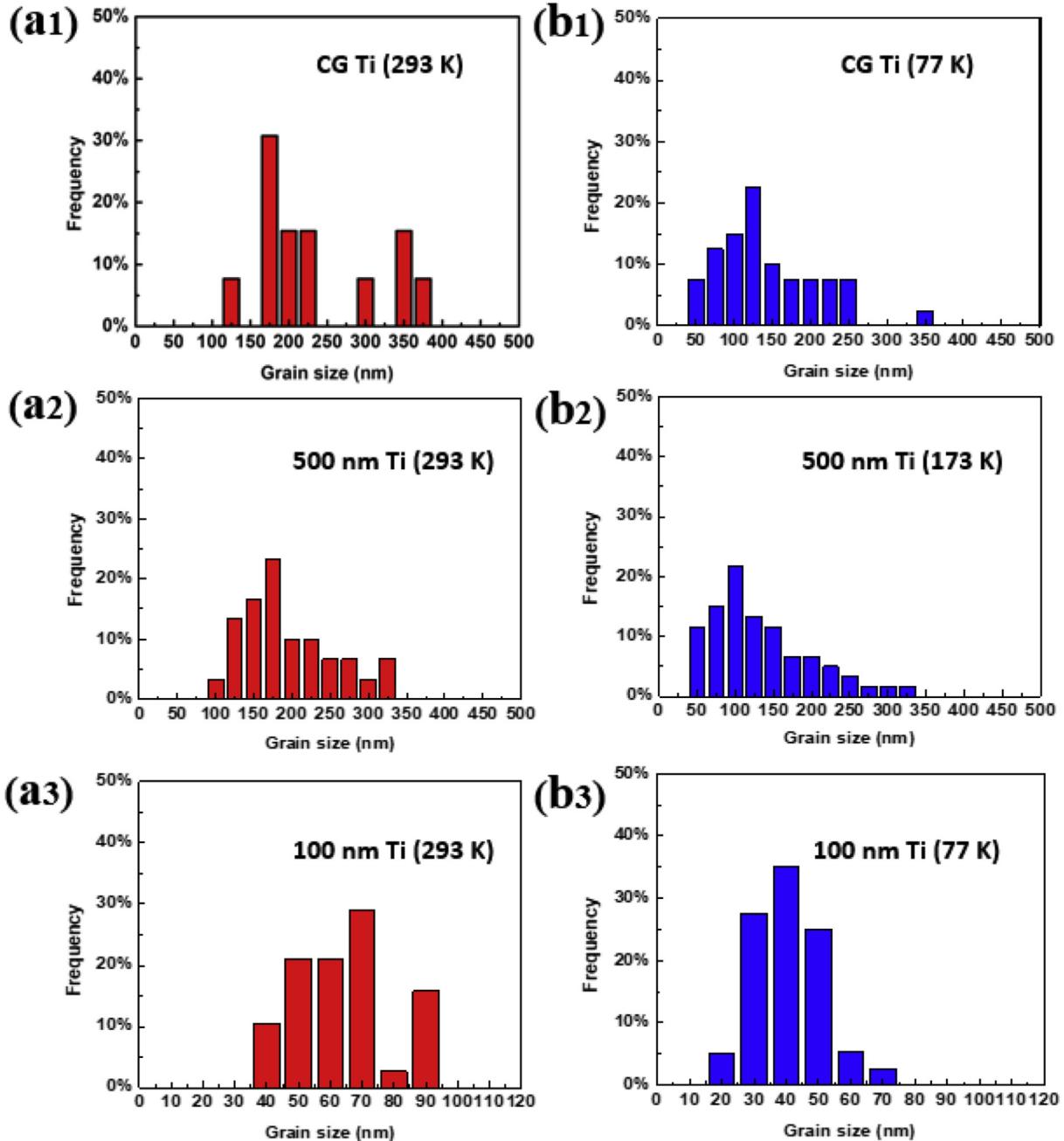
The Zener-Hollomon parameter [71],  $Z$ , was initially used to describe the relationship between flow stress, strain-rate, and temperature:  $\ln Z = \ln \dot{\epsilon} + \frac{Q}{RT}$ . The strain-rate inside the shear band is approximately  $10^5 \text{ s}^{-1}$ , the activation energy for deformation is 150 kJ, and the temperature rise inside the shear band is assumed to be 500 K, the recrystallization temperature for Ti. Mishra et al. [23], Derby [72], and Li et al. [73] found that the recrystallized grain size is proportional to the value  $\ln Z$ :

$$d_R(\ln Z)^B = A \quad (18)$$

By incorporating (18) into (17) and assuming the width of the ellipsoid equal to the size of recrystallized grains ( $W = d_R$ ), one can obtain the critical dislocation density for shear localization for titanium:

$$\rho_c = \left[ \frac{3e}{4} \left( \frac{(\ln Z)^B}{A} \right) \right]^2 \quad (19)$$

where the fitting parameters  $A$  and  $B$  for CG and UFG 500 nm

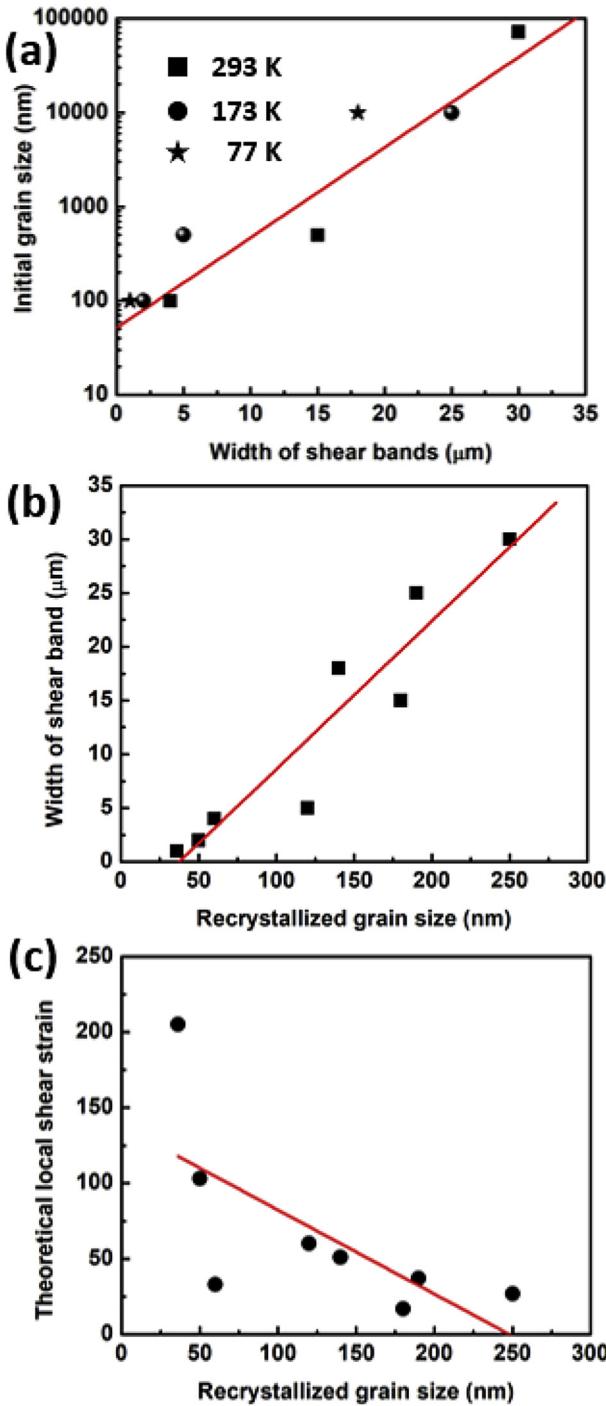


**Fig. 9.** Grain-size distribution in shear bands in CG titanium at (a1) 293 K and (b1) 77 K. Grain-size distribution in shear bands in UFG 500 nm titanium at (a2) 293 K and (b2) 173 K. Grain-size distribution in shear bands in UFG 100 nm titanium at (a3) 293 K and (b3) 77 K.

titanium are summarized in Table 2. These parameters also incorporate the effect of shear strain which can, at some point, be separated into another component. Fig. 11(b1,b2) show the predicted recrystallized grain size  $d_R$  and the critical dislocation density for shear localization for CG and UFG 500 nm titanium under different ( $\ln Z$ ) values. Once the critical dislocation density is established, by assuming the initial dislocation density of  $6 \times 10^{12} \text{ m}^{-2}$ , one can obtain the parameters  $k_h$  and  $f_s$  through the classic Taylor equation [74] ( $\tau = \tau_0 + \alpha_0 G b \sqrt{\rho}$ , where  $\tau_0$  is the yield shear stress,  $\alpha_0$  is a constant taken as 0.09,  $G = 45 \text{ GPa}$  is the shear modulus, and  $b$  is the Burgers vector taken as 0.289 nm). Table 3 shows other parameters for the Kocks-Mecking theory. It can be seen that the critical dislocation density increases with decreasing

grain size, leading to a decrease in  $W$  (as predicted from Eqn. (17)) and a consequent decrease in recrystallized grain size. The same trend is observed for the decrease in initial deformation temperature. Fig. 11 (a1,a2) show that the rate of change of dislocation density first increases and then decreases gradually to zero.

Once the subgrain configuration forms (with a width  $W$ ), it tends to break into segments which rotate to accommodate further deformation. Meyers et al. [9] showed that the recrystallized grain size of CG titanium inside the shear band could not reach the nanocrystalline region assuming static migrational recrystallization during the cooling of the shear band, thus demonstrating that the migrational recrystallization mechanism cannot account for the formation of recrystallized grains inside the shear band.



**Fig. 10.** (a) Width of shear band as a function of initial grain size. (b) Recrystallized grain size as a function of shear band width. (c) Recrystallized grain size as a function of estimated local shear strain.

**Table 2**

Parameters for the relationship of Zener-Hollomon parameter [71] with the average recrystallized grain size for titanium.

	A	B
CG Ti	1.51*10 <sup>-6</sup>	2.47
UFG 500 nm Ti	6.96*10 <sup>-7</sup>	3.65

Thus, Meyers et al. [9–11], and Hines and Vecchio et al. [60] proposed that a rotational dynamic recrystallization had to occur to account for the observed grain size refinement inside the shear band. As calculated using Eqn. (2), the temperature inside the shear bands can rise to the recrystallization temperature (between 0.4  $T_m$ –0.5  $T_m$ ) for both CG and UFG titanium. Once the elongated subgrains are achieved, they further break up into subgrains. According to the rotational dynamic recrystallization mechanism, the formation of a new equiaxed grain requires a final step: local grain-boundary segments to tilt about 30° within the deformation process [11]. The time needed for these processes can be expressed as follows [11]:

$$t = \frac{WkTf(\theta)}{4\delta_g\eta D_{b0} \exp(-Q_b/RT)} \quad (20)$$

where  $t$  is time;  $W$  is the average grain diameter ( $=d_R$ );  $\delta_g$  is grain-boundary thickness;  $\eta$  is the grain-boundary energy;  $D_{b0}$  is a constant related to grain boundary diffusion;  $Q_b$  is the activation energy for grain boundary diffusion;  $\theta$  is the subgrain misorientation.  $f(\theta)$  is given in Ref. [11]:

$$f(\theta) = \frac{3 \tan(\theta) - 2 \cos(\theta)}{3 - 6 \sin(\theta)} + \frac{2}{3} - \frac{4\sqrt{3}}{9} \ln \frac{2 + \sqrt{3}}{2 - \sqrt{3}} + \frac{4\sqrt{3}}{9} \frac{\tan(\theta/2) - 2 - \sqrt{3}}{\tan(\theta/2) - 2 + \sqrt{3}} \quad (21)$$

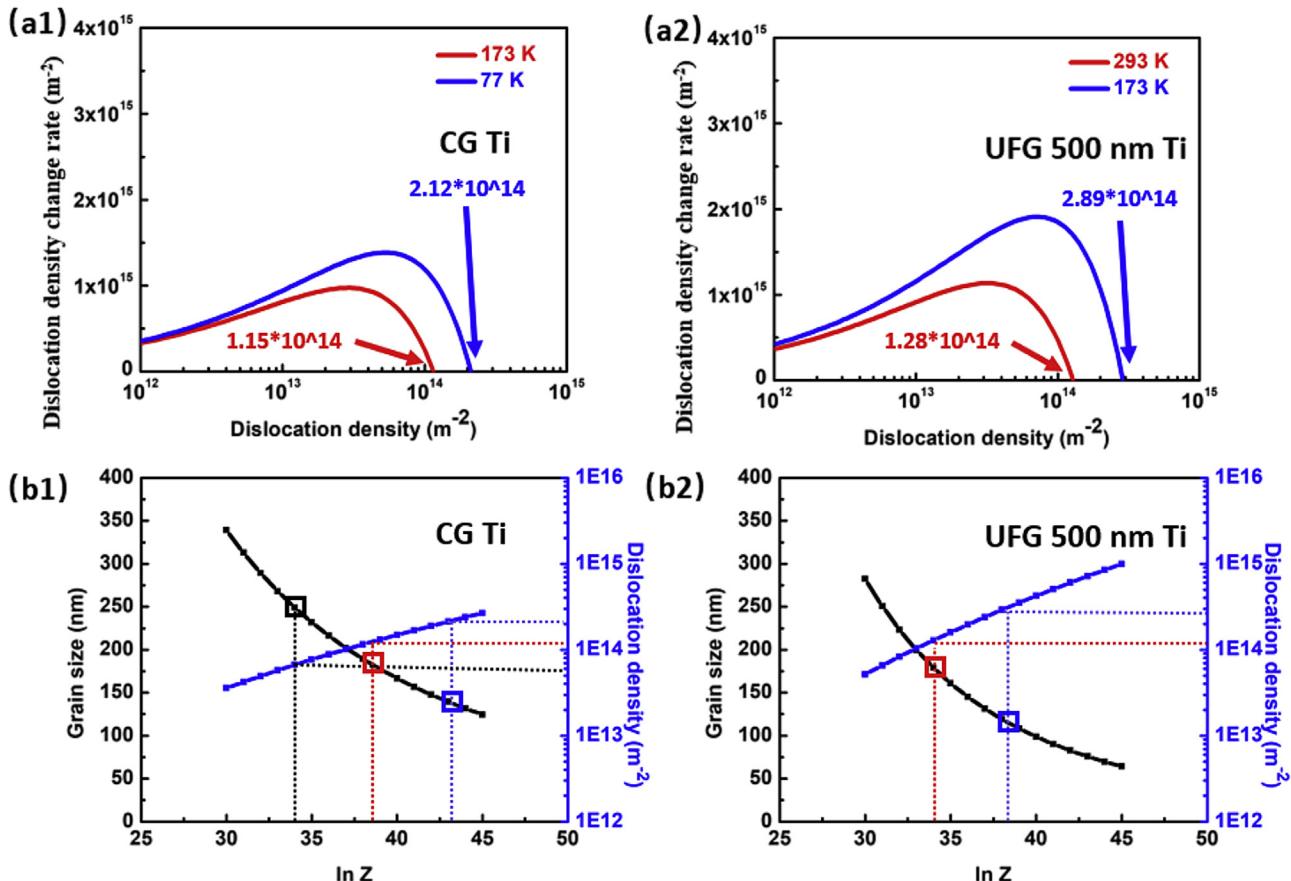
For UFG titanium,  $\delta_g = 1.5 \times 10^{-9}$  m,  $\eta = 0.675$  J/m<sup>2</sup>,  $D_{b0} = 1.4 \times 10^{-3}$  m<sup>2</sup>/s,  $Q_b = 151$  kJ/mol,  $k = 1.38 \times 10^{-23}$  J/K,  $R = 8.314$  J/(K·mol), and  $T = 800$  K [9,27]. The kinetic curves for the rotational dynamic recrystallization mechanism in a shear band can be obtained by integrating the parameters in Equations (20) and (21). Fig. 12 shows that a grain boundary can rotate 30° within 6 μs for a grain size of 200 nm; for a grain size of 40 nm, the time is reduced to 2 μs since the diffusion path is shortened. The total deformation time in the hat-shaped specimen is ~200 μs. Thus, there is ample time for the boundary segments to reorient to minimize their energy and create an equiaxed configuration during deformation. The schematic drawing showing the dynamic recrystallization process of UFG 100 nm is presented in Fig. 13(a–c). With the increase of shear deformation, the initial grain size breaks down into smaller domains. Once the low-angle grain boundaries form, the grains tend to rotate by  $\theta$  to form equiaxed grains due to energy gradients.

### 3.5. Cooling of the shear bands

It is important to establish the cooling times for the shear bands with different thickness. Assuming a constant rate of heat generation in the thin shearing zone, the solution of the heat equation for the hat-shaped specimens can be reduced to [75]:

$$T(x, t) = \frac{\delta(T_m - T_0)}{\sqrt{4\pi\chi t}} e^{-(x-R_i)^2/4xt} \left( R_i - \frac{\delta}{2} < x < R_i + \frac{\delta}{2} \right) \quad (22)$$

where  $T_0$  and  $T_m$  are the initial temperature and the maximum temperature within the shear band after the deformation;  $t$ ,  $\delta$  and  $R_i$  are the cooling time, the width of shear bands and the distance from the center of shear band to the center of the hat-shaped specimen;  $\chi$  is the heat diffusivity.  $\Delta T = T_m - T_0 = 500$  K is assumed for each shear band and  $x$  is equal to  $R_i$  in the middle of shear band. The fast cooling rates of shear bands at room and low temperatures shown in Fig. 14 are responsible for retaining the ultrafine- and nano-grain size inside the shear band and not



**Fig. 11.** (a1,a2) Rate of dislocation density change with the variation of dislocation density. (b1,b2) Relationship between critical dislocation density and subgrain size with Zener-Hollomon parameter ( $\ln Z$ ).

**Table 3**

Constants for the Kocks-Mecking model [69] and critical dislocation densities for shear localization.

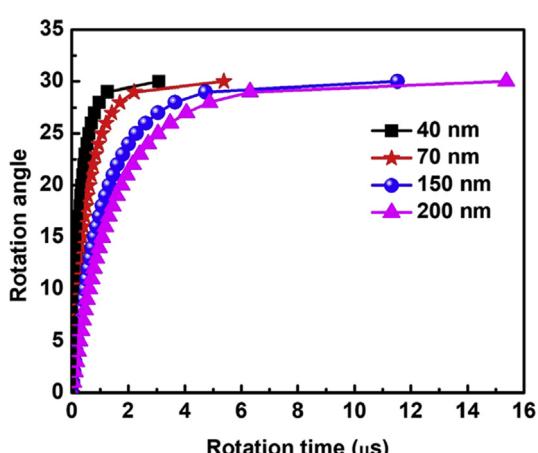
Parameters	$k_{\text{forest}}$	$f_{\text{recovery}}$	Critical dislocation density ( $\text{m}^{-2}$ )
CG Ti (173 K)	$3.63 \times 10^8$	33.8	$1.15 \times 10^{14}$
CG Ti (77 K)	$3.81 \times 10^8$	26.2	$2.12 \times 10^{14}$
500 nm Ti (293 K)	$4.01 \times 10^8$	35.4	$1.28 \times 10^{14}$
500 nm Ti (173 K)	$4.50 \times 10^8$	26.5	$2.89 \times 10^{14}$

allowing the grains to grow during cooling. Although there are differences among the different initial conditions, the cooling to ambient temperature occurs within 20  $\mu\text{s}$  for most specimens. These calculations confirm that the grains inside the shear band are formed by the rotational dynamic recrystallization process, and not by static migrational recrystallization during cooling.

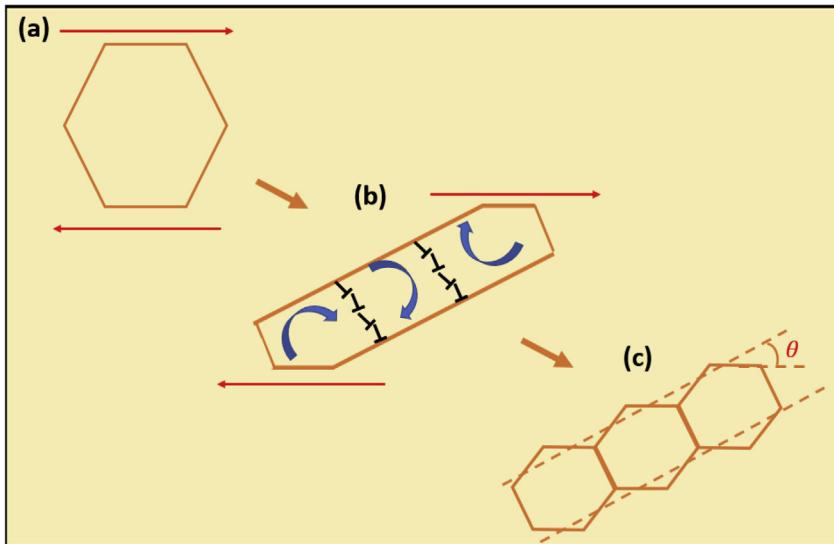
#### 4. Conclusions

Taken as a whole, the mechanical behavior and adiabatic shear band formation of CG and UFG titanium at room and cryogenic temperatures were revealed and several new findings were made which can be summarized as follows:

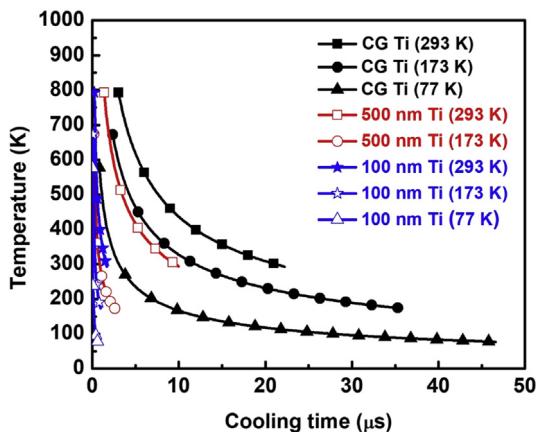
- (1) The strength of UFG titanium is significantly higher compared with CG titanium due to the Hall-Petch effect. In comparison with CG titanium, the strain hardening rate of the UFG titanium is significantly lower due to the reduced dislocation activation volume and larger incidence of grain boundaries which act as dislocation sinks. The strain-rate sensitivity of UFG titanium does not change significantly with temperature, in comparison with CG titanium, indicating no significant change of deformation mechanism.
- (2) The width of the shear band of CG titanium does not change too much with decreasing temperature. In contrast, the width of shear band of UFG 500 nm titanium decreases from  $\sim 15 \mu\text{m}$  formed at room temperature to  $\sim 5 \mu\text{m}$  at low temperature. The width of shear band of UFG 100 nm titanium decreases to  $\sim 1 \mu\text{m}$  at low temperature, which is one quarter



**Fig. 12.** Rotation of grain-boundary segments as a function of time at 800 K for grain sizes of 40 nm, 70 nm, 150 nm and 200 nm.



**Fig. 13.** Schematic drawing showing the shear-rotational dynamic recrystallization mechanism for UFG 100 nm to form recrystallized equiaxed nanograins. (a) Original ultrafine-grains; (b) formation of low-angle grain boundaries and subsequent grain rotation under the applied shear stress; (c) formation of recrystallized equiaxed nanograins through grain-boundary rotation by an angle  $\theta$ .



**Fig. 14.** Temperature in the middle of the shear bands as a function of cooling time for CG and UFG titanium [75].

of that ( $\sim 4 \mu\text{m}$ ) formed at room temperature. The combined effects of the decrease of thermal conductivity, the decrease of specific heat capacity and the increase of thermal softening for UFG titanium lead to decreased widths according to Bai-Dodd's and Grady's predictions and are indeed experimentally observed. The experimental results fall within the region bound by the two theories and show a strength sensitivity slightly higher than them; the values are closer to the Bai-Dodd theory for lower strength levels (CG) and approach the Grady theory for higher strength (UFG) Ti.

(3) A dislocation-based Kocks-Mecking model is proposed to describe the dislocation evolution prior to the dynamic recrystallization process for CG and UFG 500 nm titanium. The dislocation density increases gradually until it reaches the critical value determined by a balance in dislocation generation and annihilation. The randomly distributed dislocations form elongated ellipsoid configurations which, once the temperature reaches the recrystallization value ( $\sim 0.4 T_{\text{melting}}$ ), break down through subgrain rotation. It is shown that the width of the sub-grains decreases with increasing dislocation density.

- (4) The formation of nanograins inside the shear bands is explained in terms of the rotational dynamic recrystallization mechanism, which is expanded to incorporate the differences observed for the different initial conditions (grain size, initial deformation temperature, and shear strain).
- (5) A reduction in the recrystallized grain size inside the shear bands for both CG and UFG titanium is observed for the initial deformation temperature in the cryogenic region. This is caused by a higher dislocation density reached at the onset of recrystallization due to higher accumulation rates in the first stage of deformation, at sub-ambient temperatures.
- (6) The Zener-Hollomon parameter was used to predict the recrystallized grain size and critical dislocation density for shear localization. Additionally, the larger local shear strain in the shear bands at cryogenic temperatures, because of the decrease of width, also contributes to a more severe plastic deformation, inducing smaller grain size inside the shear band.

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