Real time correlation between flow stress and dislocation density in steel during deformation

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ABSTRACT

We performed *in situ* compression of interstitial-free steel nanoblades in a transmission electron microscopy (TEM) in order to determine the relation between the evolution of the dislocation structures and the flow stress during deformation. In the early stage of deformation, the sample deforms elastically with a few dislocation motions. The dislocation multiplication processes have been discussed. Remarkable plastic softening with increasing dislocation density is observed after the maximum stress is reached, which can be understood as a situation in which the dislocation density is the dominant factor affecting the softening based on the Johnston-Gilman model.

Keywords: In situ nanoindentation; interstitial-free steel; plastic softening; dislocation; Johnston-Gilman model.

1. Introduction

The evolution of dislocation structures during plastic deformation is of great importance because of its relationship to the deformation mechanisms and mechanical properties of metals.

Recently, plasticity at small scales is attracting increasing attention since it has been found that many mechanical properties at the sub-micro/nanometer scale differ from those at the continuum scale [1-7]. Because of the large free surface areas presented in the small-scale samples, the competition between dislocation formation/multiplication and escape of dislocations from the surface will lead to an extremely complex response during deformation in contrast to that of a bulk sample. For example, the dislocation escape rate can overwhelm the dislocation formation rate so that the dislocation starvation or dislocation-nucleation controlled mechanism [8-13] is dominant in face-centered cubic (fcc) nano/micropillars. However, the situation is more complicated in the case of body-centered cubic (bcc) nano/micropillars. Several factors, such as the sample geometry, initial dislocation density and strain rate etc. may play an important role in determining the deformation mechanism in bcc crystals [14-19]. Under a critical size, they might behave like fcc nano/micropillars, in which the dominating factor is the dislocation starvation mechanism [16-18]. On the other hand, because of differences in the dislocation mobility and dislocation core structure, dislocation multiplication might be prolific in the larger pillars [15-17]. Furthermore, which model is prevailing during the evolution of plasticity, i. e. conventional forest hardening/Taylor hardening [20] or softening [15] as described by Johnston-Gilman [21, 22] has not been well investigated since most of the attentions have been focused on the size effect at the early stage of deformation such as yielding. Direct correlation between flow stress and dislocation density is necessary for further understanding the plasticity and to provide experimental evidence for simulation. In a previous work [18], we found that the dislocation starvation mechanism is the dominant deformation mechanism in an Fe-Si nanopillar of about 100 nm in diameter. In the present paper, we report dislocation-density-dominant behavior in a larger interstitial-free steel sample, in which dislocations undergo multiplication while plastic

softening was observed after the stress reaching the maximum value. We still use scanning TEM (STEM) for our *in situ* observations, and by changing the sample into a nanoblade shape, both easy observation of the dislocation motion, as in a thin film sample, and easy calculation of the stress, as for a pillar sample, can be achieved at the same time.

2. Experimental

The material used is Ti-added interstitial-free steel. The yield stress and maximum stress of the bulk sample after the tensile test are around 100 MPa and 250 MPa [23], respectively. A grain with a size about 100 μm after heat treatment was used to fabricate nanoblades with a focused ion beam (JEM-9320 FIB). The specimen surface was coated with carbon before FIB milling to avoid damage due to the incident Ga^+ ions. The final beam current used was 10 pA with an accelerating voltage of 30 kV. The nanoblade is about 500 nm in width, 110 nm in thickness, and 600 nm in length. The compression test was performed with a Hysitron PicoIndenter at room temperature in a JEOL 2010F TEM using a diamond flat-end punch with a diameter of 2.5 μm . The system was operated in displacement-control compression mode with a typical strain rate of $\sim 1.0 \times 10^{-3} \text{ s}^{-1}$. The resulting movies (10 x playback) can be found in the supplementary information, as movie-P1 (sample P1) and movie-P2 (sample P2). The compression direction was along the $[3\bar{3}2]$ direction. The $[\bar{1}11](1\bar{1}2)$ slip system has the maximum Schmid factor of 0.43. The other two $[111](2\bar{3}1)$ and $[11\bar{1}](3\bar{2}1)$ slip systems have relatively smaller Schmid factors of 0.24. The direction of the incident electron beam was along the $[121]$ zone axis, and the movie was recorded in STEM mode at a frame rate of 30 frames/s. After the in-situ compression test, the dislocation character was determined by conventional TEM contrast analysis and TEM trace analysis (JEOL 2000FX and 2010F).

3. Results and discussion

3.1 Structure evolution during the *in situ* deformation

Fig. 1(a) shows the true stress-true strain curves of the two nanoblades captured during the *in situ* deformation. The stress was calculated from the measured loads over the cross-sectional area at the top of the nanoblades, assuming that the plastic volume of the specimen was conserved during deformation. Both curves exhibit elastic behavior in the low strain range. After reaching a maximum value of about 1.1 GPa, the flow stresses gradually decrease under further strain, similar to the curves as described in Ref. [15] with supplementary movie 2B. The maximum stress obtained with this small sample is much higher than that of the bulk counterpart (about 250 MPa), which indicates the existence of a size effect in the test material. Work hardening is limited after the elastic loading, unlike the obvious work hardening for the deformed Mo pillars in Ref. [1] and pure Fe in Ref. [24]. We will discuss this difference in detail later. The fluctuations or oscillation in the stress are presumably associated with the multiplication and the depletion of the mobile dislocations.

Figs. 1(b)-(f) are STEM snapshots showing the microstructure of nanoblade P1 extracted from the video at the instances marked as b–f in Fig. 1(a), respectively. The indenter comes from the left side of the image and compresses the sample in the horizontal direction in the image. The protecting carbon layer is in between the indenter and the nanoblade, as indicated in Fig. 1(b). Before 27 s, no new dislocations form, but an as-grown dislocation remains in the left upper corner after the FIB milling, giving a dislocation density $\sim 3 \times 10^{12} \text{ m}^{-2}$. A newly formed dislocation appears at 27.3 s, as indicated by the white triangle in Fig. 1(b). As shown in Fig. 1(c), many dislocations have formed before the stress reaches the maximum level. The projection direction of the Burgers vector $[\bar{1}11]$ is indicated by the white arrow in the image. It is obvious

that at this early stage, the sample has undergone elastic as well as microplastic deformation, but not pure elastic deformation. It is noticed that most of the dislocations formed in the early stage have a screw character, especially at the sample top and middle (see dislocation character analysis in Fig. 4). However, the dislocations formed at the sample bottom are more of a mixed feature. It is speculated that at the sample bottom, the bulk root may partly constrain the movement of the newly formed half loop dislocation. The screw segment may undergo cross-slip until the edge part can escape the sample surface. That's why the half loop bow out from the sample root has longer time duration with mixed feature. However, the half loop nucleated away from the sample root can move freely without constraints. The edge segments move faster than the screw ones and escape the nanoblade easily, leaving the remained dislocations mainly of a screw type. The details of the dislocation structure evolution prior to 74 s are discussed in the next section. The dislocation density can be calculated by a line-intercept method, where, the dislocation density is the number of points (N) divided by the total line-length of random lines, L_T , multiplied by foil thickness, t , ($\rho = \frac{N}{L_T t}$) [25]. The dislocation density was calculated to be about $1.5 \times 10^{14} \text{ m}^{-2}$ in Fig. 1(b). Several seconds later, another type of dislocations along the projection direction of the Burgers vector $[11\bar{1}]$ can be observed. Some junctions formed at the lower part of the nanoblade due to the interaction between the two types of dislocations. The drop in the stress at about 92 s is presumably attributed to the generation and motion of collective dislocations (Fig. 1(e)). A dislocation density $\sim 9 \times 10^{15} \text{ m}^{-2}$ was measured at 92 s. As indicated by white triangle, the visible dislocations that pile up at the bottom of the sample become wavy and tangled, together with lots of junctions caused by cross-slips and mutual dislocation interactions. In Fig. 1(f), the area without dislocation contrast at the left upper corner

has gradually increased. The reason for this is related to the sample rotation or a change in the crystallographic orientation, which are commonly observed during small-scale deformation [22-25].

3.2 Dislocation multiplication in the early stage

In the early stage, the dislocation density is low, thus the formation and multiplication of dislocation is distinguishable. Here we will discuss two types of dislocation multiplication process in nanoblade P1. One type of multiplication is the formation of dislocation loops or segments originating from a pre-existing (as-grown) dislocation, which are shown in detail in Figs. 2 and 3. The other type of multiplication is the formation of new dislocations from the sample root, as indicated by the white triangle in Fig.1(b). The detail of dislocation multiplication process became much complex at higher strain range. For example, it was frequently observed that several dislocations simultaneously appear in the nanoblade during the small pop-in or stress fluctuation, but it is hard to say whether they are forming from the same source or undergo self-multiplication process (referring to movie-P1 at about 7 s).

Figs. 2 and 3 show magnified snapshots of the dislocation multiplication process originating from the as-grown dislocation before 74 s (Fig. 1(c)). Fig. 2(e) is a sketch of the dislocation shown in Figs. 2(a)–(d). The as-grown dislocation is divided into four segments, which are labeled 1–4 in Fig. 2(a). Segments 1 and 3 are relatively straighter than segments 2 and 4, and the movement of the curved dislocation segments 2 and 4 can be observed easily. In Figs. 2(b) and (c), segment 4 changes into a sharp diamond shape by cross slipping. At the same time, the repeated cross slipping of segment 2 (see movie-P1) results in a shorter dislocation line but a larger curvature. Within 0.03 s (Fig. 2(d)), the dislocation segments 1 and 3 instantaneously disappear and segment 4 evolves into a diamond-shaped loop labeled 4'. Meanwhile, a new

dislocation loop, 2', as well as a segment, A, form. The possible evolution process that occurs within this short period (0.03s) is shown in Fig. 2(f). The curved as-grown dislocation is divided into four segments and labeled 1-4. In step I, it is supposed that these four dislocation segments may sit on different slip planes. During the next step, segments 1 to 3 bow outward on their respective slip planes. These three segments further expand in step III, and sections B and E in segments 1 and 3, as indicated by arrows, may reach the sample surface and escape the sample. The remaining sections, C and D, come in contact and form a loop with dislocation 2. At the same time, the remaining section, F, forms a loop with dislocation 4, and segment A remains on the slip plane. Through this process, the single as-grown dislocation multiplies into two loops and a segment. These loops and segment act as dislocation sources in the following deformation, as shown in Fig. 3, where several dislocations multiply at the sample top (left region). In Fig. 3(a), dislocation loop 2' gradually expands and evolves into a longer dislocation line within 0.04 s, as indicated by the arrow in Fig. 3(b). Several seconds later, due to the escaping of the edge segment, dislocation loop 4' evolves into a long dislocation line as indicated by arrow in Fig. 3(c). Further multiplication processes can be observed clearly in movie-P1.

Another type of dislocation source lies at the sample root at about 3 s in the movie-P1, as indicated by the triangle in Fig. 1(b). The multiplication of dislocations located at the sample root may be introduced by the large stress concentration at the sample root or surface roughness. Damages caused by the FIB milling may also provide some dislocation sources. Several dislocations have formed at the sample root in Fig. 1(c). If we assume this dislocation source to be a Frank-Read one, its length estimated from the distance between the anchoring points visible in Fig. 1(b) is ~ 86 nm. A dislocation source with a size, L , of 86 nm corresponds to a shear

stress τ , [26] with $\tau = \frac{Gb}{L} = \frac{80 \cdot 0.25}{86} \approx 0.23$ GPa, where G is the shear modulus and b ($= 0.25$

nm) [27] is the magnitude of the Burgers vector. Meanwhile, the flow stress at 27 s is about 0.5 GPa, as determined from the stress-strain curve. Thus, the shear stress τ for Frank-Read dislocation nucleation (taking the maximum Schmid factor of 0.43) is $0.50 \times 0.43 \approx 0.22$ GPa (about $G/400$), which is consistent with the value estimated from the dislocation structure.

3.3 Dislocation structure after deformation

Fig. 4(a) is a STEM image showing the overall dislocation structure in the deformed nanoblade sample P2. The region of interest is indicated by the broken-line rectangle; the dislocation density in this area appears to be locally low. Figs. 4(b)-(d) are bright field images taken under different diffraction vectors near $[111]$ axis. Fig. 4(e) is a schematic of the dislocation lines. Dislocation lines A, B, and C are visible under the diffraction vector $\mathbf{g} = [\bar{1}01]$, as shown in (b). Dislocation A is out of contrast under $\mathbf{g} = [\bar{1}10]$ (Fig. 4(c)) and dislocations B and C are out of contrast under $\mathbf{g} = [01\bar{1}]$ (Fig. 4(d)). Provided that the Burgers vectors of these dislocations are of the $\frac{1}{2}\langle 111 \rangle$ type, the Burgers vector of dislocation A can be deduced to be $\frac{1}{2}[\bar{1}1\bar{1}]$, and those of dislocations B and C to be $\frac{1}{2}[\bar{1}11]$. For ease of understanding, the projection directions of the Burgers vectors $[\bar{1}1\bar{1}]$ and $[\bar{1}11]$ are also indicated in Figs. 4(c) and (d), respectively. It is clear that long, straight screw dislocations are predominantly formed in the deformed pillar, because almost all the dislocation lines are parallel to one of the Burgers vectors. In general, the slip plane can be identified by the cross product of the Burgers vector and the dislocation line vector of the dislocation. A rough estimation of the dislocation line vector by trace analysis suggests that the line vector of dislocation A is nearly parallel to $[6, 7, \bar{1}6]$. It is

possible that the slip plane of dislocation A is $(\bar{1}10)$, since the cross product of the Burgers vector, $[11\bar{1}]$, and the dislocation line vector, $[6,7,\bar{1}6]$, is $(\bar{9},10,1)$. On the other hand, the line vectors of dislocations B and C are almost parallel to their Burgers vectors, $[\bar{1}11]$. In that case, the slip plane(s) for dislocations B and C cannot be uniquely determined. However, since dislocations A and B/C do not lie in the same slip plane, (101) , the intersections between dislocations A and B/C ought to be of the jog type.

3.4 Discussion

The results of our investigation shed light on several phenomena that are associated with the relationship between dislocation structures/motions of metals and their mechanical behavior. From the stress-strain curve and the observed dislocation motion of the sample tested, we found that the dislocation nucleation in the small-scale sample in the early stage may not be directly connected with large pop-in excursions as was observed in the bulk sample [23]. In the present study, it is found that the stress fluctuation is small during the formation of a single dislocation, such as that formed at 27.3 s in Fig. 1(b). The much smoother curves in the present study also differ from the other bcc crystals tested with smaller sample size [18, 24], which have lots of obvious pop-ins. It is supported that the sample size may play an important role in determine the microstructure evolution during the deformation in bcc crystals. In addition, although the shear stress of $G/400$ calculated for a Schmid factor of 0.43 is in good agreement with the stress necessary to operate a Frank-Read source, it is much smaller than the theoretical stress of $G/2\pi$. It is thought that the larger sample size relative to that in our former experiment may be the main reason for the small calculated stress [18]. Furthermore, the FIB-fabricated sample may provide

a lot of dislocation sources, so that the dislocation formation may not necessarily require a stress as high as the theoretical stress.

As we have mentioned that the strain hardening in the present test is limited, differing from other bcc crystals [1, 24]. Although the initial defects density is thought to have an effect on the stress evolution [15, 19], it is hard to compare our results with other researches since detailed defects density is not available and seldom research has touched this topic. According to the simulation in Ref. [15] with movie 2B which has a number of initial dislocations, the sample size may be the main reason for this softening but not the initial dislocation density. Furthermore, our investigation, as shown in Fig. 1(d), is in good agreement with the simulation [15] where the stress drop concurs with the formation of dislocation junctions. The strain softening associated with dislocation multiplication provided a clear experimental evidence as predicted in Ref. [15]. Although it is believed that jogs on a screw dislocation can impede the gliding of dislocation, that is, its glide resistance can increase, resulting in strain hardening. It seems that in the present study this effect is not strong enough to affect the plastic softening. However, the dislocation mobility and mobile dislocation density effect may play an important role. To understand this, Johnston and Gilman model [21, 22, 26] was revisited. The sample orientation, sample geometry and aspect ratio [28] may affect the stress evolution but need further investigation. The dislocation velocity $\bar{v}$ can be related to the applied shear stress through a relation given by [21, 22, 26]:

$$\bar{v} \propto \tau^m, \quad (1)$$

where m is the dislocation velocity-stress exponent. The strain rate $\dot{\gamma}$ can be defined on the basis of the dislocation theory as follows [29]:

$$\dot{\gamma} = \rho b \bar{v}, \quad (2)$$

where b is the magnitude of the Burgers vector. Since the strain rate is almost steady/constant under the displacement-control compression mode, we can combine the two equations as follows:

$$\dot{\gamma} \propto \rho b \tau^m, \quad (3)$$

Then, we can rewrite the equation as:

$$\log \tau \propto \frac{1}{m} \log \frac{\dot{\gamma}}{b} - \frac{1}{m} \log \rho \quad \text{or} \quad \log \tau \propto A - B \cdot \log \rho \quad (4)$$

$$\text{or} \quad \frac{\partial \tau}{\partial \ln \dot{\gamma}} \propto \frac{\tau}{m} \quad (5)$$

where $A = \frac{1}{m} \log \frac{\dot{\gamma}}{b}$ is constant and B is $\frac{1}{m}$. At the very beginning of deformation, the dislocation density is low. The dislocations are free to glide but cannot move fast enough at low stresses, as indicated by Eq. (1). Therefore the stress in the nanoblade rises to produce a sufficient elastic strain in the specimen. As it does so, the dislocations multiply rapidly. The multiplication of dislocations continues as the strain increases, producing more dislocations than required. As a result, the stress drops and plastic softening is observed. According to Eq. (4), the relation between stress and dislocation density in nanoblade P2 for a strain larger than 0.12 can be plotted as shown in Fig. 5, where the decrease in stress with an increase in dislocation density is very clear. The m value calculated from the linear fit (broken line) is approximately 7, which is close to the value of about 5.5 [30] calculated from strain-rate cycling data for high-purity iron in bulk sample. It is suggested [30] that at low velocities, there is sufficient thermal assistance and time to allow for the dislocation tangle formation and the development of long-range internal

stress. The dislocation cross-slips and dislocation tangles observed in our experiment may be further evidence for this low m value. According to Aono et al, [31] τ is about 20 and $\frac{\partial \tau}{\partial \ln \dot{\gamma}} = 3$ for pure iron at room temperature. So, m can be deduced to be ~ 7 based on Eq. (5), in good consistent with our measurement. It is speculated that the m value might relate to the intrinsic lattice friction to a screw dislocation motion and the nature of the core structure of it [31]. A much higher m value of ~ 40 for edge dislocation was reported for Fe-3.25% Si [32] at room temperature. It is supported that the difference in dislocation mobility between screw and edge dislocation can result in big difference in m value. Since most of the dislocations observed in the present study are of the screw type which have a mobility lower than that of the edge dislocations observed in the Fe-3.25 % Si [32], the low m value is reasonable.

On the basis of the results obtained in this study, we can also cast light on the structural changes induced during the deformation of a bulk sample by treating the nanoblade sample as a grain in a bulk sample. As is already known, the as-grown dislocations can act as dislocation sources. In the case of the small-scale sample, some parts of the as-grown dislocation have escaped the sample, but some segments remain. It is clear that, owing to this slipping process, the dislocation density can increase greatly. Accordingly, in the bulk sample, an as-grown dislocation may slip to the grain boundary during deformation. In addition, a few segments may sink into the grain boundary while a few segments may remain in the grain. These remaining segments may then become dislocation sources. At the same time, the grain boundaries may also act as a dislocation source in the bulk sample. The newly formed dislocations may bow out from the grain boundary and multiply, as was noticed in the nanoblade sample. The multiplication of dislocations at these sources may lead to an increase in the dislocation density and eventually

result in stress softening, which can be understood as a situation in which the dislocation density is the dominant factor affecting the softening based on the Johnston-Gilman model.

4. Summary

It was found that, during the *in situ* compression of an IF steel nanoblade fabricated by FIB milling, dislocation formation is activated at a stress level much lower than the expected theoretical stress. With further straining, the stress gradually increases with increasing dislocation density until a maximum stress is reached. Further increases in the dislocation density lead to stress softening which can be understood well based on the Johnston-Gilman model.

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References

- [1] S. Brinckmann, J.-Y. Kim, J. R. Greer, Fundamental differences in mechanical behavior between two types of crystals at the nanoscale. *Phys. Rev. Lett.* 100 (2008) 155502.
- [2] D. Kiener, W. Grosinger, G. Dehm, R. Pippan, A further step towards an understanding of size-dependent crystal plasticity: In situ tension experiments of miniaturized single-crystal copper samples. *Acta Mater.* 56 (2008) 580-592.
- [3] M. D. Uchic, D. M. Dimiduk, J. N. Florando, W. D. Nix, Sample dimensions influence strength and crystal plasticity. *Science* 305 (2004) 986-989.
- [4] S. H. Oh, M. Legros, D. Kiener, G. Dehm, In situ observation of dislocation nucleation and escape in a submicrometre aluminium single crystal. *Nat. Mater.* 8 (2009) 95-100.
- [5] Z. W. Shan, J. Li, Y. Q. Cheng, A. M. Minor, S. A. S. Asif, O. L. Warren, E. Ma, Plastic flow and failure resistance of metallic glass: Insight from in situ compression of nanopillars. *Phys. Rev. B* 77 (2008) 155419.
- [6] A. M. Minor, J. J. W. Morris, E. A. Stach, Quantitative in situ nanoindentation in an electron microscope. *Appl. Phys. Lett.* 79 (2001) 1625-1627.
- [7] T. Ohmura, A. M. Minor, E. A. Stach, J. W. M. Jr., Dislocation–grain boundary interactions in martensitic steel observed through in situ nanoindentation in a transmission electron microscope. *J. Mater. Res.* 19 (2004) 3626-3632.
- [8] D. Kiener, P. J. Guruprasad, S. M. Keralavarma, G. Dehm, A. A. Benzerga, Work hardening in micropillar compression: In situ experiments and modeling.
- [9] W. D. Nix, J. R. Greer, G. Feng, E. T. Lilleodden, Deformation at the nanometer and micrometer length scales: Effects of strain gradients and dislocation starvation. *Thin Solid Films* 515 (2007) 3152-3157.

- [10] J. R. Greer, W. C. Oliver, W. D. Nix, Size dependence of mechanical properties of gold at the micron scale in the absence of strain gradients. *Acta Mater.* 53 (2005) 1821-1830.
- [11] J. R. Greer, W. D. Nix, Nanoscale gold pillars strengthened through dislocation starvation. *Phys. Rev. B* 73 (2006) 245410.
- [12] D. Kiener, A. M. Minor, Source-controlled yield and hardening of Cu(1 0 0) studied by in situ transmission electron microscopy. *Acta Mater.* 59 (2011) 1328-1337.
- [13] D. Kiener, A. M. Minor, Source Truncation and Exhaustion: Insights from Quantitative in situ TEM Tensile Testing. *Nano. lett.* 11 (2011) 3816-3820.
- [14] C. R. Weinberger, W. Cai, Surface-controlled dislocation multiplication in metal micropillars. *Proc. Natl. Acad. Sci. USA* 105 (2008) 14304-14307.
- [15] I. Ryu, W. D. Nix, W. Cai, Plasticity of BCC micro-pillars controlled by competition between dislocation multiplication and depletion. *Acta Mater.* 61 (2013) 3233–3241.
- [16] L. Huang, Q.-J. Li, Z.-W. Shan, J. Li, J. Sun, E. Ma, A new regime for mechanical annealing and strong sample-size strengthening in body centred cubic molybdenum. *Nat. Commun.* 2 (2011) 547.
- [17] C. Chisholm, H. Bei, M. B. Lowry, J. Oh, S. A. S. Asif, O. L. Warren, Z. W. Shan, E. P. George, A. M. Minor, Dislocation starvation and exhaustion hardening in Mo alloy nanofibers. *Acta Mater.* 60 (2012) 2258-2264.
- [18] L. Zhang, T. Ohmura, K. Sekido, T. Hara, K. Nakajima, K. Tsuzaki, Dislocation character transition and related mechanical response in a body-centered cubic single crystal. *Scripta Mater.* 67 (2012) 388-391.
- [19] H. Bei, S. Shim, G. M. Pharr, E. P. George, Effects of pre-strain on the compressive stress–strain response of Mo-alloy single-crystal micropillars. *Acta Mater.* 56 (2008) 4762-4770.

- [20] J. R. Greer, C. R. Weinberger, W. Cai, Comparing the strength of f.c.c. and b.c.c. sub-micrometer pillars: Compression experiments and dislocation dynamics simulations. *Mater. Sci. Eng. A* 493 (2008) 21-25.
- [21] W. G. Johnston, J. J. Gilman, Dislocation velocities, dislocation densities, and plastic flow in lithium fluoride crystals. *J. Appl. Phys.* 30 (1959) 129-144.
- [22] W. G. Johnston, Yield points and delay times in single crystals. *J. Appl. Phys.* 33 (1962) 2716-2730.
- [23] K. Sekido, T. Ohmura, L. Zhang, T. Hara, K. Tsuzaki, The effect of interstitial carbon on the initiation of plastic deformation of steels. *Mat. Sci. Eng. A* 530 (2011) 396-401.
- [24] K. Y. Xie, S. Shrestha, Y. Cao, P. J. Felfer, Y. Wang, X. Liao, J. M. Cairney, S. P. Ringer, The effect of pre-existing defects on the strength and deformation behavior of α -Fe nanopillars. *Acta Mater.* 61 (2013) 439-452.
- [25] D. M. Norfleet, D. M. Dimiduk, S. J. Polasik, M. D. Uchic, M. J. Mills, Dislocation structures and their relationship to strength in deformed nickel microcrystals. *Acta Mater.* 56 (2008) 2988-3001.
- [26] D. Hull, D. J. Bacon, Introduction to dislocations -third edition, Butterworth heinemann, Oxford, 1984.
- [27] W. W. Gerberich, J. C. Nelson, E. T. Lilleodden, P. Anderson, J. T. Wroblek, Indentation induced dislocation nucleation: the initial yield point. *Acta Mater.* 44 (1996) 3585-3598.
- [28] Z. F. Zhang, H. Zhang, X. F. Pan, J. Das, J. Eckert, Effect of aspect ratio on the compressive deformation and fracture behaviour of Zr-based bulk metallic glass. *Phil. Mag. Lett.* 85 (2005) 513-521.
- [29] M. Zaiser, Scale invariance in plastic flow of crystalline solids. *Adv. Phys.* 55 (2006) 185 -245.
- [30] J. T. Michalak, The influence of temperature on the development of long-range internal stress during the plastic deformation of high-purity iron. *Acta Metall.* 13 (1965) 213-222.

- [31] Y. Aono, E. Kuramoto, K. Kitajima, plastic deformation of high-purity iron single crystals. Reports of Research institute for Applied Mechanics XXIX (1981) 127-193.
- [32] D. F. Stein, J. R. Low, Mobility of edge dislocations in silicon iron crystals. J. appl. Phys. 31 (1960) 362-369.

Figure captions:

Fig. 1 (a) True stress-true strain curves of the two nanoblades captured during *in situ* deformation. (b)-(f) STEM snapshots extracted from the video showing the microstructure of nanoblade P1 at the instances marked as b–f in (a), respectively. All images were obtained along the $[121]$ zone axis. The indenter is located at the left side of the image. The protecting carbon layer is in between the indenter and the nanoblade, as indicated in (b). The projection directions of the Burgers vectors $[\bar{1}11]$ and $[11\bar{1}]$ are indicated in (c) and (d), respectively.

Fig. 2 (a)-(d) Magnified STEM images extracted from the video corresponding to formation of the dislocation loop in nanoblade P1 before 50 s. (e) Sketches of the dislocations as shown from (a) to (d); (f) Schematic showing one possible process.

Fig. 3 Magnified STEM images extracted from the video corresponding to the dislocation multiplication from the loops in nanoblade P1 after 50 s.

Fig. 4(a) STEM image showing the overall dislocation structure in deformed nanoblade P2. (b)-(d) Bright-field images of the area indicated by the square in (a). The diffraction vector $\mathbf{g}$ near the $[111]$ axis is indicated in the right corner of the image. For ease of understanding, the projection directions of Burgers vectors $[\bar{1}11]$ and $[11\bar{1}]$ are also indicated in (c) and (d), respectively. Scale bars in (b)-(d) represents 50 nm. (e) Schematic showing the three dislocations.

Fig. 5 Relationship between stress and dislocation density in nanoblade P2 after the maximum stress is reached at a strain of 0.12.

Figure 1

Figure 2

Figure 3

Figure 4

(a)

(b)

(c)

(d)

(e)

Figure 5