

Crystalline Deformation in the Small Scale

Murat Demiral, Anish Roy and Vadim V. Silberschmidt

Abstract In the last two decades, experimental observations demonstrated—and numerical simulations confirmed—that plastic deformation in the small scale, i.e. at the micron or sub-micron scales, is different from that at the macro-scale; this phenomenon is known as *size effect*. It was observed mostly in indentation, torsion and bending experiments, being ascribed to strong gradients of strain in such deformation processes. The size effect was also reported in uniaxial micro- and nano-pillar compression experiments in spite of their inherent lack of (or limited) macroscopic strain gradients. In the present study, we first review some critical and essential experimental studies that were conducted over the years to analyse various mechanisms that govern deformation in the small scale. In the second part, different modelling approaches describing this phenomenon are briefly reviewed.

1 Introduction

Modern high-technology applications such as medical devices, thermal barrier coatings, micro- and nano-electro mechanical systems and semiconductors as well as gems industry increasingly use components with sizes down to a few micro-metres and even smaller. The respective manufacturing processes can thus include

M. Demiral (✉)

Department of Mechanical Engineering, University of Turkish Aeronautical Association,
06790 Ankara, Turkey
e-mail: mdemiral@thk.edu.tr

A. Roy · V.V. Silberschmidt

Wolfson School of Mechanical and Manufacturing Engineering,
Loughborough University, Loughborough, Leicestershire LE11 3TU, UK
e-mail: A.Roy3@lboro.ac.uk

V.V. Silberschmidt

e-mail: V.Silberschmidt@lboro.ac.uk

forming and shaping at a small scale. Plastic deformations in such a scale are known to differ from those at the macro-scale, resulting in a so-called ‘size effects’.

Understanding deformation mechanisms and assessing mechanical performance at small scales are necessary to realize the full potential of emerging nano- and micro-technologies and to design new products with superior characteristics. Also, knowledge of links between the microstructural and geometrical factors can provide additional ways to enhance component performance and clarify their design constraints.

The phenomena of size effect can be separated into two groups—“intrinsic” and “extrinsic”. Intrinsic size effects are related to strengthening due to microstructural constraints such as grain size and second-phase particles and have been studied by a materials science community for more than a century. Extrinsic size effects arise from dimensional constraints, e.g. due to a sample size and were discovered in the last two decades; hence, they are currently a topic of rigorous investigations [1].

An extrinsic size effect was mostly observed in indentation, torsion and bending experiments, where it was ascribed to a higher dislocation density due to the generation of geometrically necessary dislocations (GNDs), which, in turn, led to strong gradients of strain in a deformation process. To understand the underlying physics for this phenomenon, uniaxial compression experiments of surface-dominated micro-pillars, eliminating the effect of strain gradients, were performed. However, the size effect was still reported, with the ultimate tensile strength and yield strength scaling with an external sample size in a power-law fashion, sometimes attaining a significant fraction of material’s theoretical strength. Several different explanations were proposed in the literature to elucidate it.

Alongside with experimental studies, various numerical simulations were used to investigate the size effect. Though they are in many cases less cumbersome to implement than detailed test at microscale, these methods have their own limitations. For instance, quantum and atomistic simulations cannot be performed on realistic time scale and structures while classical continuum plasticity theories cannot explain the dependence of mechanical response on size as no length scale enters the constitutive description. Multi-scale continuum theories incorporating spatial gradients and/or volume integrals accounting for non-local interactions provide an alternative for this.

The purpose of the present review is to survey crystalline deformation in the small scale. Both experimental and numerical applications of size effects are briefly reviewed.

2 Experimental Observations

In this section we review some critical and essential experimental studies that were conducted over the years and paved the way for theoretical and modelling work to characterise and study various mechanisms that govern deformation in the small scale.

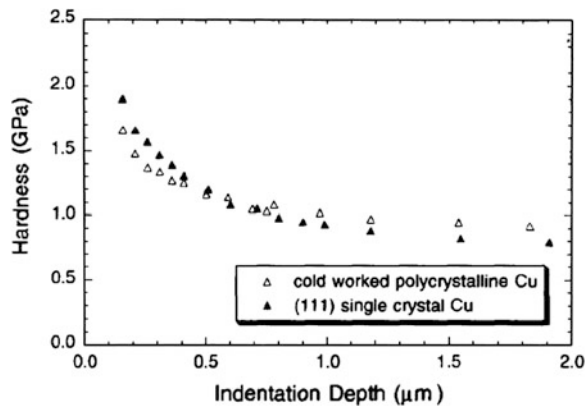
2.1 Indentation Size Effects

Experiments carried out in the past few decades demonstrated strong indentation size effects (ISEs) in crystalline materials. Stelmashenko et al. [2] and [3] reported that the measured hardness values were observed to increase with a decreasing indentation size, especially in the sub-micrometre depth regime. Figure 1 demonstrates a typical ISE observed in single-crystal copper and a cold worked polycrystalline copper.

This observation is not surprising as hardness measurements have been recognized to be size-dependent since early experiments by Tabor [4]. The research into the ISE gained prominence partly motivated by the development of large-scale application of thin films in electronic components and partly by the availability of novel methods of probing mechanical properties in very small volumes. Different physical and geometric mechanisms were suggested for ISE. Some of the proposed mechanisms or factors are: (1) a pile-up or a sink-in of the deformed surface during indentation; (2) a loading rate of the indenter; (3) a grain size, or the Hall-Patch effect; (4) presence of an oxidation layer or a work-hardened surface layer and (5) a geometric effect of indenter's tip (e.g. its radius) [5].

Some authors argued that the size dependence of material's mechanical properties resulted from an increase in strain gradients inherent to localized zones, which necessitated the presence of geometrically necessary dislocations resulted in additional hardening [6]. The characteristic features of deformation in metals are formation, motion and storage of dislocations. According to a Taylor's hardening rule, dislocation storage is responsible for material hardening. Stored dislocations can be divided into two groups: statistically stored dislocations (SSDs) and GNDs. SSDs are generated by trapping each other in a random way and believed to be dependent on effective plastic strain. On the other hand, GNDs are the stored dislocations that relieve plastic-deformation incompatibilities within the material caused by non-uniform dislocation slip. The density of GNDs is directly proportional to the gradients of effective plastic strain, and their presence causes additional storage of defects and,

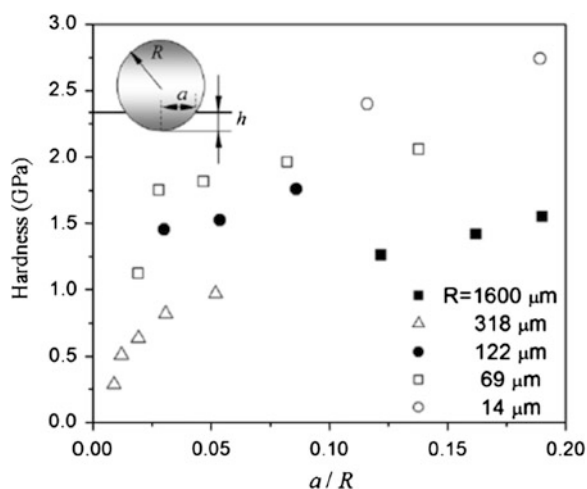
Fig. 1 Indentation size effect in crystalline materials. Reprinted with permission from [3]



hence, increases the deformation resistance by acting as obstacles to SSDs [6]. The dependency of SSDs and GNDs, respectively, on the effective plastic strain and its gradients is responsible for the size effect. The smaller the length scale, the larger the density of GNDs relative to that of SSDs and, consequently, the higher the plastic strain gradients compared to average plastic strains. An important and often misunderstood caveat is that SSDs and GNDs are essentially identical and simply labelled differently for convenience. Such an approach inevitably leads to the requirement for additional phenomenological laws with non-physical parameters to account for the interaction between GNDs and SSDs (e.g. in the form of annihilation and generation rules). A physically reasonable and accurate model of continuum dislocation mechanics should account for all dislocations consistently. Such an approach was proposed by Acharya [7] and developed to model most of the well-known benchmark problems in small-scale plasticity [8, 9].

Different indenter types were also used to study ISE. For instance, Qu et al. [10] used five different spherical indenter tips with radius ranging from 14 to 1600 μm to measure hardness. It was observed that indentation hardness H increased with an increase in the ratio of contact radius a to the indenter radius R (Fig. 2). Swadener and George [11] also observed that H increased continuously with an increase in the indentation depth using a spherical indenter, i.e., the so-called *reversed size effect*, contrary to the usual size effect observed in sharp indentation. The reverse dependence of hardness on indentation depth is explained by different dislocation densities underneath the indenters. In the case of a sharp indenter, the average density of SSDs is independent of the indentation depth, while the density of GNDs is inversely proportional to it (Fig. 3a). On the other hand, in the case of a spherical indenter, although the density of SSDs increases with an increase in the indentation depth (through a contact radius) the density of GNDs becomes essentially independent (Fig. 3b). The total dislocation density, therefore, displays a reversed depth dependence for sharp and spherical indenters; so does the magnitude of indentation

Fig. 2 Effect of ratio of contact radius (a) to spherical indenter radius (R) on indentation hardness of iridium. Reprinted with permission from [10]



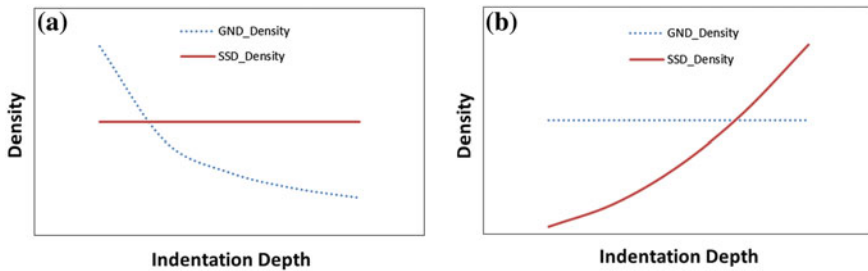


Fig. 3 Schematic plot of variations in GND and SSD densities with indentation depth using a sharp (a) and spherical indenter (b)

hardness [5]. On the other hand, the experimental study of [12] with a sharp indenter demonstrated that the total GND density below the indents decreased with decreasing indentation depth. This observation contradicts the strain-gradient theories attributing size-dependent material properties to GNDs. Abu Al-Rub and Voyiadjis [13] interpreted the size effect in hardness experiments with a spherical indenter in a different way. It was explained that to get ISE in spherical indentation similar to that in sharp indentation, plastic strain should be independent of the sphere size to provide independence of plastic strain on indentation depth. In that study, the hardness values obtained with different spherical indenters were compared for the same a/R ratio representing the same plastic strain value. A decrease in hardness with an increasing spherical indenter size was reported, which can be also interpreted based on Fig. 2.

2.2 Size Effect in Pillar Compression

Recently, Uchic and co-workers [14] developed a testing methodology to measure the flow behaviour of miniature samples in compression. In this test micro-scale cylindrical samples fabricated using a focused-ion-beam (FIB) milling technique (for instance Fig. 4a) were uniaxially compressed in a nano indentation system equipped with a flat punch [15]. This method was specifically designed to probe mechanical properties as a function of the decreasing sample size. Later this technique was extended by [16] to perform uniaxial compression tests on Au nano-pillars.

Uchic et al. [14] tested three different materials with a pillar size from 1 to 40 μm : Ni, Ni₃Al-1 %Ta and a Ni-based super alloy with a (269) orientation. These experiments showed an increase in the strength of crystals by up to 15 times compared to bulk Ni with a decrease in the sample size. However, no significant increase in the work-hardening rate of the crystals was observed. It was also reported that frequent strain bursts—avalanches of dislocations—and finite discrete

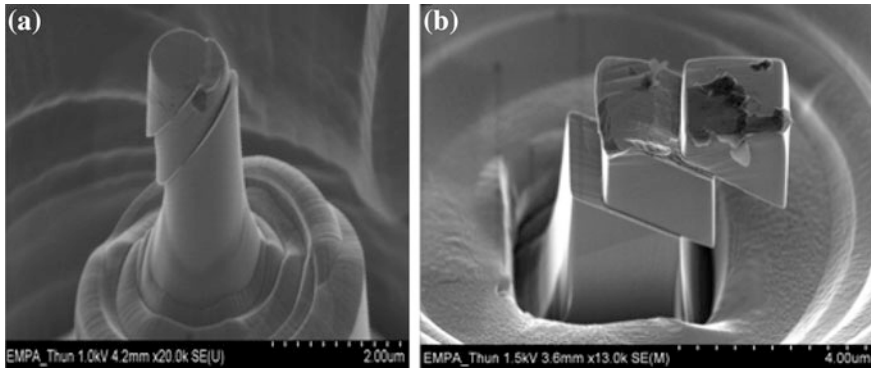
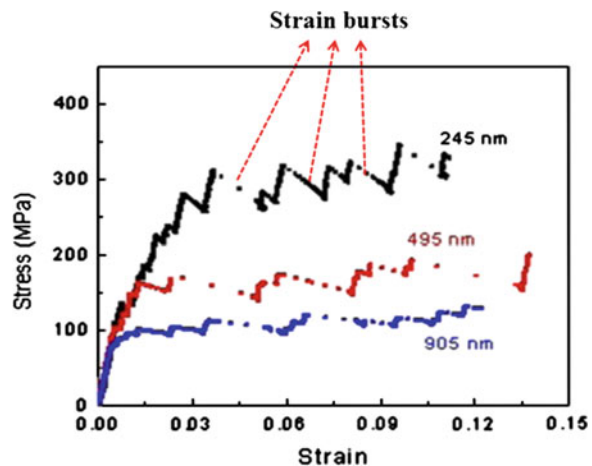


Fig. 4 SEM images of Ti-15-333 single-crystal micro-pillars with circular (a) and square (b) cross sections after compression

slip bands along the gauge length of the crystal were observed when its size diminished. In contrast to a smooth stress-strain curve obtained during compression of a bulk material, the stress-strain curves of small-size pillars contained several discrete strain bursts, with strain jumping discontinuously to an increased value while the stress value remained constant or decreased. Figure 5 demonstrates typical strain bursts, more frequent in small-size pillars, observed on a stress-strain curve of f.c.c. nano pillars. Greer and Nix [17] performed similar tests on (001)-oriented Au crystals with dimensions of less than 1 μm . The study showed a trend of an increase in the strength of the pillar with a decrease in its size. It was also revealed that the flow-stress value of ~ 800 MPa was observed in compression of 200 nm Au specimens, which was extraordinarily high compared to its bulk counterpart with a corresponding value around 25 MPa at 10 % strain.

Fig. 5 Typical stress-strain curves in compression of Au pillars with various diameters. Reprinted with permission from [34]



Recently, Kiener et al. [18] reported a new method to measure tensile behaviour of single-crystals at the micro- and nano-scales using the samples fabricated with FIB. The mechanical test was performed inside a scanning electron microscope (SEM) and a transmission electron microscope (TEM). In this experiment, f.c.c. Single-crystal Cu samples with diameters ranging from 500 nm to 8 μm with an aspect ratio ranging from 1 to 13.5 were tested. This study revealed the presence of a size effect, which strongly depended on the sample's aspect ratio; however, the reported size effect was less pronounced than its compression counterpart. It was reported that size-dependent hardening was linked to dislocation pile-ups due to their constrained glide in the sample. Jennings and Greer [19] performed an in situ tensile-deformation test with (111)-oriented single-crystal Cu nano-pillars with diameters ranging from 75 to 165 nm fabricated with e-beam lithography and electroplating. A power-law relationship between strength and diameter of nano-pillar was reported similar to that for fabricated with FIB machining. In that study, inhomogeneous deformation was explained by annihilation of dislocations at free surfaces of the pillar.

Most studies of size effects deal with f.c.c. Materials with only recent studies investigating b.c.c. Crystalline materials. In the following section we discuss studies in f.c.c. and b.c.c. crystals. The relevant reviews for more complex microstructures such as non-cubic single-crystals, shape memory alloys, nano-crystalline metals, nano-laminate composites and metallic glasses are available elsewhere [1] and the references therein).

2.2.1 Face-Centred Cubic Metals

Several mechanisms have been proposed for deformation of nano- and micron-size f.c.c. pillar structures. Among the suggested theories “hardening by dislocation starvation” is the mostly accepted one to explain the size effects in f.c.c. nano-pillars. The basic premise of the theory is as follows: Owing to smaller dimensions of the nano-size pillars, dislocations present at the onset of plastic deformation leave the specimen before they multiply. Having reached this “dislocation starvation” state, new dislocations are necessary to nucleate both at the sample surface and in the bulk of the crystal in the course of deformation since the movement of dislocations is required for compatible plastic deformation. As the nucleation of these new dislocations requires high stresses approaching near-theoretical strength, the strength of nano-size pillars is observed to increase with a decreasing sample size. This theory appears to be in good agreement with in situ TEM performed by [20]. Figure 6a demonstrates the FIB micro-fabricated (111) Ni pillar with 160 nm of diameter in this study. It is apparent that there is a high initial dislocation density in the pillar. However, the pre-existing dislocations progressively leave the pillar during the initial compression stage, and a completely dislocation-free pillar is finally obtained (Fig. 6b). Computational atomistic simulations performed by different research groups also justify this theory [21].

A prominent deformation mechanism for micron-sized pillars (larger than 500 nm) is described in terms of a single-arm source theory proposed and

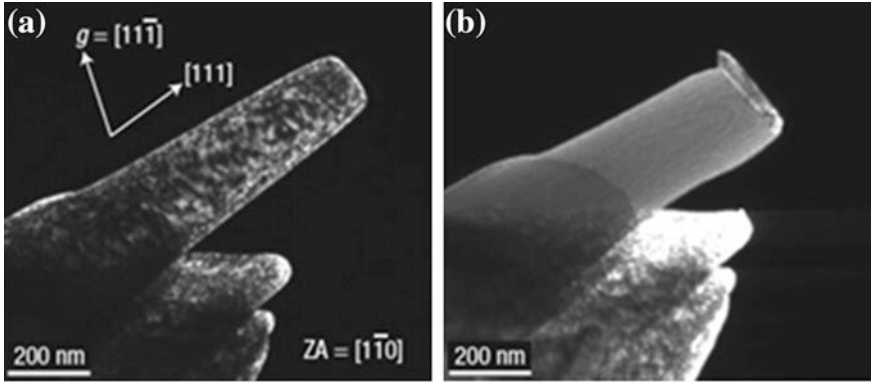


Fig. 6 Dark-field TEM image of Ni pillar before tests (a) and after first compression (b). Reprinted with permission from [20]

developed by [22]. In this theory, creation of dislocations occurred as a result of operation of partial Frank-Read sources or truncated sources also known as *single-arm sources*. This approach suggested that a random distribution of dislocation sources either existed initially in pillars or generated by interaction of initially present dislocations, and the average source strength τ_s was related with the average source length λ by the following equation:

$$\tau_s = k_s \mu \frac{\ln(\lambda/b)}{(\lambda/b)}, \quad (1)$$

where k_s is a source-hardening rate, μ is the shear modulus and b is the Burgers vector. According to this equation, with a decrease in the diameter of pillar, the source length became smaller, hence overall strength increased. Ng and Nyan [23] studied the size effect of Al micro-pillars with diameters ranging from 1.2 to 6 μm either coated or centre-filled with a tungsten-based compound. A higher strain-hardening rate and a much smoother stress-strain curve suggested suppression of dislocation avalanches and a lack of nucleation-controlled plasticity. The TEM analysis revealed higher dislocation densities in the post-deformed specimens confirming the trapping of dislocations inside the micro-pillars rather than annihilating at the free surface in contrast to nano-sized pillars [1].

Maass and co-workers studied the role of strain gradients on plastic deformation of micron-size pillars using a synchrotron micro-diffraction technique [24]. These studies demonstrated that geometrically necessary dislocations were generated during the deformation process leading to strain gradients. The assumed dislocation starvation theory, therefore, was not applicable to micron-size pillars since it was impossible to squeeze all the dislocations to the surface of the pillar. Likewise, [25] argued that although deformation in a pillar-compression test was macroscopically homogenous, it was heterogeneous microscopically, i.e. GNDs vanished macroscopically but were presented locally. Akarapu et al. [26] numerically demonstrated

that deformation was heterogeneous from its onset and was confined to the discrete slip bands. On the other hand, SEM images of micron-sized pillars taken after their compression in [27] suggested that deformation evolved in an inhomogeneous manner, though it was applied homogeneously since the end regions of the pillars appeared non-deformed while the sample's centre was sheared by multiple slip zones.

The FIB technique is currently the most prevalent method for producing small-size pillars to investigate the influence of sample's dimensions on mechanical properties. In this method Ga^+ ions are bombarded and implanted to fabricate the pillars. This process inevitably introduces surface dislocation loops, and precipitates also instigate surface amorphization. The presence of FIB-induced defects led several researchers to infer that the fabrication process may play a significant role in the observed size effects. On the one hand, [28] proposed that increasing strength of pillars with a decrease in their size would be a consequence of the increased volume fraction of a FIB-damaged layer with a decreasing pillar diameter. Kiener et al. [29] investigated Ga^+ ion-induced damage with TEM and Monte Carlo simulations, where a non-negligible influence of the ion damage in the order of 100 MPa (assuming Taylor hardening) was reported for submicron-sized samples. In order to understand importance of the fabrication technique on the size effects, [30] developed a FIB-less method to produce nano-pillars. In this method, arrays of vertically oriented gold and copper nano-pillars were created based on patterning polymethylmethacrylate with electron beam lithography and subsequent electroplating into the prescribed template. In this technique, pillars were produced intentionally with non-zero dislocation densities to compare and contrast them with those produced with the FIB method; otherwise, pillars without dislocations would render theoretical strengths regardless of size. That study demonstrated that nano-size pillars created without Ga^+ bombardment and containing initial dislocations with density comparable with that for pillars created with Ga^+ bombardment exhibited an identical size effect with the FIB-produced pillars. The study evidently suggested that the observed size effect in small-size pillars was a function of microstructure rather than the fabrication technique.

2.2.2 Body-Centred Cubic Structures

Experimental and computational studies of compression of single-crystal Mo nano-pillars showed that the deformation mechanism in b.c.c. nano-pillars was fundamentally different from that of f.c.c. nano-pillars. Brinckmann et al. [31] reported a comparison of uniaxial compression results for Mo and Au nano-pillars. That study revealed that size-dependent strengths, stochastic discrete bursts in stress-strain curves, fractions of attained theoretical strength as well as extents of strengthening were different. These discrepancies were attributed to profound differences in the plasticity mechanisms of f.c.c. and b.c.c. metals as also confirmed by atomistic simulations [32]. In b.c.c. metals, glide of screw components of a dislocation loop is not restricted to any particular single plane and can also cross-slip

on any other favourable crystallographic plane during shearing of the crystal, whereas the edge components glide on a specific plane. In b.c.c. metals mobility of screw dislocations is estimated at around one fortieth of the motion of edge dislocations. The dislocations in b.c.c. nano-pillars, therefore, are likely to have a higher residence time inside a pillar rather than leaving it, significantly increasing the probability of interaction of individual dislocations, their multiplication and formation of junctions, which, in turn, lead to new dislocation sources. As the junction size is proportional to the size of the pillar, the junctions are shorter for small-size b.c.c. pillars. It is therefore harder to break through the gliding mobile dislocations leading to an increase in strength of the pillar. On the other hand, as the increase in temperature greatly increases mobility of screw dislocations, the number of formed junctions is reduced requiring nucleation of new dislocations. The size effect, therefore, in b.c.c. metals would be expected to become closer to that in f.c.c. metals at higher temperatures. However, the b.c.c. pillars never become dislocation starved as in the f.c.c. nano-pillars; instead, a complex networks of short dislocation segments are formed [32]. This is also consistent with molecular-dynamics (MD) and dislocations-dynamics (DD) simulations of [33].

Kim and Greer [34] reported size-dependent strengths of (001) oriented Mo nano-pillars in compression and tension. The observed higher flow stresses were explained by an increase in yield strength rather than strain-hardening. It was also reported that the amount of strain-hardening under tension was much lower compared to that under compression demonstrating a tension-compression asymmetry. This observed asymmetry was attributed to the differences in the Peierls stress, i.e. lattice resistance to dislocation motion, in twinning and anti-twinning direction. It is worth mentioning that friction stress or intrinsic lattice resistance, accounting for the flow-stress indirectly related to dislocation activities, is negligibly small in f.c.c. metals but it is significant in b.c.c. metals [35]. In [34], TEM images of Mo nano-pillars with diameter of 100 nm before and after the deformation were analysed. The results indicated formation of an entangled dislocation substructure and justified differences in the deformation behaviour of f.c.c. and b.c.c. metals. The effect of crystal orientation on the tension-compression asymmetry of Mo nano-pillars was also studied by Kim and Greer [34]. It was shown that compressive flow stresses were higher in (001) orientation and smaller in (011) orientation compared to tensile flow-stresses. This study revealed that tension-compression asymmetry was a function of size of the pillar for sizes less than 800 nm, whereas strength for larger pillars approached size-independent bulk values.

The influence of applied strain-rate on the size effect in b.c.c. single crystals was investigated by [36]. Under a constant displacement rate, the imposed strain-rate varied by around one order of magnitude between the largest and smallest Mo pillar ranging from 200 to 800 nm. In that study, differences in the yield stress of around 50 % for the smallest and around 20 % for the largest pillars were reported. The study revealed that the strain rate had an influence on the observed size effects in nano-size pillars. Kim et al. [36] also studied different b.c.c. metals in addition to Mo. (011)-oriented single-crystal nano-size pillars of Ta, W, Nb and Mo were subjected to uniaxial compression and tension. A power-law for size-dependent

flow-stress of pillars was found together with size-dependency of observed tension-compression asymmetry. However, the authors did not observe any consistent correlation between the strain-hardening exponent and the material's type or size. That implied that strain-hardening was more likely to be a function of the initial microstructure rather than the pillar size, whereas the yield and flow-stress fundamentally depended on the size of nano-pillar [1].

The deformation mechanism in micron-sized b.c.c. pillars is similar to that in nano-sized pillars, unlike the different mechanism in f.c.c. nano- and micro-pillars. Therefore, the mechanisms explained above for b.c.c. single-crystal nano-pillars can be generalized to micron-size pillars.

In summary, it is justified that in b.c.c. single-crystals a single dislocation can generate multiple new dislocations. Dislocation segments will further interact and form Frank-Read sources leading to an increase in dislocation density and requiring significant levels of flow stress due to inherent characteristic of screw dislocation gliding at different slip planes. The hardening mechanism of b.c.c. single crystals via entanglement of dislocation segments is similar to the forest-hardening model in bulk crystal plasticity [31].

3 Modelling Approaches

3.1 Models Developed for ISE

A classical continuum plasticity theory cannot explain the size dependence of mechanical response as no length scale enters the constitutive description. However, strain-gradient plasticity schemes have been successful in characterizing the size effect in components subjected to inhomogeneous loading states. This success is related to the inclusion of a microstructural length-scale parameter in the governing equations for deformation. Gradient approaches typically retain terms in the constitutive equations of higher- or lower-order gradients with coefficients that characterize length-scale measures of the deformation microstructure associated with the non-local continuum [13]. Therefore, classical continuum plasticity theories including strain gradients represent a collective behaviour of dislocations and their interactions at the micro-scale. However, there exists a lower limit on the length scale, below which the continuum plasticity theories are not applicable. In other words, the continuum plasticity theories are only applicable at scales larger than spacing between individual dislocations. This is similar to the relation between the elasticity and lattice theories; where the elasticity theory is applicable to a few lattice spacing (atomic spacing), below which the lattice theory governs. Gao et al. [37] identified that the lower limit for applicability of a strain-gradient plasticity theory was around 100 nm. In other words, only when the characteristic length of deformation is larger than 100 nm, strain-gradient plasticity theories can be used.

In gradient-plasticity theories, a constitutive length-scale parameter l is used to scale the effects of strain gradients and is thought of as an internal material length, related to storage of GNDs. The strain-gradient effects become important when the characteristic length associated with deformation becomes comparable to the intrinsic material length-scale parameter l . In other words, if the representative length of non-uniform deformation is much larger than l , strain-gradient effects are negligible, and the strain-gradient theories degenerate to classical plasticity theories [37]. The study of [38] indicated that indentation experiments might be the most effective way of measuring the length-scale parameter as a typical tension test cannot be effectively used to determine the material properties of gradient theories due to uniform deformation, whereas in indentation significant work-hardening evolves due to severe and non-uniform plastic deformation and damage concentrates in the localized region directly below the indenter.

Nix and Gao [39] modelled the indentation size effect for crystalline materials. The following characteristic form explaining the depth dependence of hardness was obtained from this model:

$$\frac{H}{H_0} = \sqrt{1 + \frac{h^*}{h}}, \quad (2)$$

where H is hardness for a given indentation depth h , H_0 is hardness in the limit of infinite depth and h^* is the characteristic length depending on the indenter shape, shear modulus and H_0 . They estimated the material length scale parameter l from the micro-indentation experiments of [3] to be 12 and 5.84 μm for annealed single-crystal copper and for cold-worked polycrystalline copper, respectively. Yuan and Chen [40] proposed that the unique intrinsic material length parameter l could be computationally determined by fitting the [39] model from micro-indentation experiments, and they identified $l = 6 \mu\text{m}$ for poly-crystal copper and $l = 20 \mu\text{m}$ for single-crystal copper using the finite element (FE) method. Begley and Hutchinson [38] estimated that the material length-scale associated with the stretch gradients ranged from 1/4 to 1 μm , while the material lengths associated with rotation gradients were on the order of 4 μm by fitting micro-indentation hardness data.

Strain-gradient plasticity theories can be attributed to two frameworks: higher-order and lower-order continuum theories. The higher-order theories belong to the Mindlin's framework. In this framework, higher-order stresses are involved as a work conjugate of the strain gradient described with the following equation:

$$\delta w = \sigma'_{ij} \delta \varepsilon_{ij} + \tau'_{ijk} \delta \eta_{ijk}, \quad (3)$$

where δw is the work increment per unit volume of an incompressible solid due to a variation of displacement, σ'_{ij} is the deviatoric part of the Cauchy stress, $\delta \varepsilon_{ij}$ is the variation of strain, τ'_{ijk} is the deviatoric part of higher-order Cauchy stress and $\delta \eta_{ijk}$ is the variation of strain gradient. Here, the order of equilibrium equations is higher than that of the conventional continuum theories requiring additional boundary conditions. On the other hand, in the lower-order strain gradient plasticity theory,

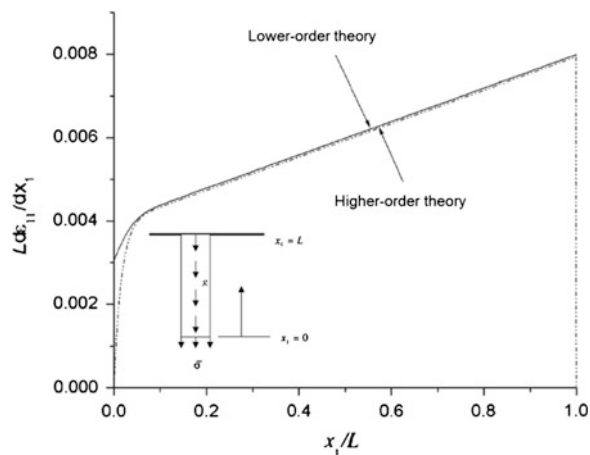
the higher-order stresses are not incorporated in the constitutive equations; hence, no additional boundary conditions are required. In this type of non-local plasticity theory, the strain gradient effects come into play through the incremental plastic modulus.

Huang et al. [41] compared the solutions obtained using higher-order and lower-order strain gradient theories for a one-dimensional hypothetical problem, where a bar was fixed at one end and subjected to a constant body force and a uniform stress at the free end along the main axis of the bar (Fig. 7). The distribution of strain gradient in the bar using lower-order and higher-order strain gradient theories is shown in Fig. 7. It is clearly visible that both theories agree very well except the area near the ends of the bar. As the higher-order stresses become significant at the boundaries, different strain-gradient distributions are obtained using different frameworks of the theories.

Various numerical simulation techniques are also used to study the underlying mechanics in indentation experiments in the small scale. For instance, deformation-induced lattice rotations below an indent have attracted attention as there exists a close connection between crystallographic shear, the main mechanism governing deformation, and the resulting lattice spin.

Some studies have attempted to characterize the observed phenomena, with the use of different experiments such as non-destructive 3D synchrotron diffraction, 3D electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM). A limited number of numerical studies attempted to analyse physical deformation mechanisms leading to lattice rotations. For instance, Wang et al. [42] demonstrated lattice rotations for a single crystal of Cu with different orientations using a 3D crystal-plasticity (CP) FE method. Zaafarani et al. [43] proposed a physically based CP model based on dislocation-rate formulations to explain likely reasons for deformation-induced patterns consisting of multiple narrow zones with alternating crystalline reorientation. However, the model consistently overestimated the extent of lattice rotations observed in experiments. Recently, Demiral et al. [44, 45] developed a

Fig. 7 Distribution of strain gradients in bar predicted by lower-order (conventional theory of mechanism-based strain gradient) and higher-order (mechanism-based strain gradient theory) theories. Reprinted with permission from [41]



3D elastic–viscoplastic enhanced modelling of strain-gradient crystal-plasticity (EMSGCP) (its details are given in Sect. 3.2) FE model for nano-indentation of Ti alloy to demonstrate the influence of strain gradients on the resulting deformation patterns. The study demonstrated that the introduction of strain gradients changed the activity of the slip systems as well as a relative contribution to the overall plastic slip. The EMSGCP theory predicted that plasticity occurred due to activity of multiple slip systems when compared to that of the CP theory.

3.2 Modelling Size Effect in Pillar Compression Experiment

Numerical modelling and simulations of pillar-compression experiments have been performed by several research teams. Among these, Zhang et al. [46] presented a parametric study of design of accurate pillar-compression experiments using 2D and 3D isotropic plasticity FE modelling. In that study, geometric factors such as the curvature at the area of pillar's connection to the substrate, the aspect ratio and taper of the pillar, misalignment between the indenter tip and the pillar, different material characteristics and plastic buckling phenomena were analysed extensively. Schuster et al. [47] followed that work and studied the effect of specimen taper on compressive strength of metallic glass. On the other hand, Chen et al. [48] focused on local stress concentration in metallic-glass pillars using an isotropic plasticity model. Raabe et al. [49] used a CP FE model to investigate the influence of stability of the initial crystal orientation, aspect ratio and contact conditions between the indenter tip and the pillar on anisotropy and changes in crystallographic orientation during the deformation. That study revealed that the evolution of orientation changes was in part due to a shape change owing to buckling rather than crystallographic orientation solely. Shade et al. [50] performed a combined experimental and CP FE study to examine lateral constraint effects on compression of single crystals. It was reported there that the degree of lateral constraint in a compression test system could influence the behaviour of the material tested.

Recently, Demiral et al. [51] proposed the EMSGCP theory, which accounts for geometric effects in addition to intrinsic properties in capturing the size effect. In the following section this theory is briefly explained.

In the EMSGCP theory, the initial strength of a slip systems ($g_T^z|_{t=0}$), i.e. the *critical resolved shear stress* (CRSS), was governed by pre-existing GNDs in the workpiece together with SSDs, i.e. $g_T^z|_{t=0} = g_S^z|_{t=0} + g_G^z|_{t=0}$. In this theory, $g_S^z|_{t=0}$ and $g_G^z|_{t=0}$ were linked with initial densities of SSDs ($\rho_S|_{t=0}$) and GNDs ($\rho_G|_{t=0}$) as

$g_S^z|_{t=0} = K\sqrt{\rho_S|_{t=0}}$, $g_G^z|_{t=0} = K\sqrt{\rho|_{t=0}}\left(\overline{S/V}\right)^2$ via the constant K , similar to the Taylor relation. The GND density term was expressed as a function of the normalized surface-to-volume ratio $\overline{S/V}$ (hence, dimensionless) for the component under study. In the study, the surface-to-volume ratio of the workpiece materials was normalized with an idealised workpiece geometry corresponding to $S/V = 1 \mu\text{m}^{-1}$.

The evolution of slip resistance during loading was the result of hardening due to the SSDs (Δg_S^α) and GNDs (Δg_G^α) on the slip system, which followed:

$$g_T^\alpha = g_S^\alpha|_{t=0} + g_G^\alpha|_{t=0} + \sqrt{(\Delta g_S^\alpha)^2 + (\Delta g_G^\alpha)^2}, \quad (4a)$$

where

$$\frac{\alpha}{S} = \sum_{\beta=1}^N h_{\alpha\beta} \Delta \gamma^\beta, \Delta g_G^\alpha = \alpha_T \mu \sqrt{b n_G^\alpha}. \quad (4b)$$

Here, $h_{\alpha\beta}$, α_T , μ , b and n_G^α correspond to the slip-hardening modulus, the Taylor coefficient, the shear modulus, the Burgers vector and the effective density of geometrically necessary dislocations, respectively.

The hardening model was used to represent $h_{\alpha\beta}$, as follows:

$$h_{\alpha\alpha} = h_0 \text{sech}^2 \left| \frac{h_0 \tilde{\gamma}}{g_T^\alpha|_{\text{sat}} - g_T^\alpha|_{t=0}} \right|, h_{\alpha\beta} = q h_{\alpha\alpha} (\alpha \neq \beta), \quad (5)$$

$$\tilde{\gamma} = \sum_{\alpha} \int_0^t |\dot{\gamma}^\alpha| dt,$$

where h_0 is the initial hardening parameter, $g_T^\alpha|_{\text{sat}}$ is the saturation stress of the slip system α , q is the latent hardening ratio, and $\tilde{\gamma}$ is the Taylor cumulative shear strain on all slip systems. The effective GND density n_G^α was given by

$$n_G^\alpha = \left| \mathbf{m}^\alpha \times \sum_{\beta} s^{\alpha\beta} \nabla \gamma^\beta \times \mathbf{m}^\beta \right|, \quad (6)$$

where s^α is the slip direction, $\mathbf{m}^\alpha$ is the slip-plane normal, $s^{\alpha\beta} = \mathbf{s}^\alpha \cdot \mathbf{s}^\beta$ and $\nabla \gamma^\beta$ is the gradient of shear strain in each slip system. To calculate $\nabla \gamma^\beta$ the scheme proposed in [44] was followed. The model was implemented in the implicit FE code ABAQUS/Explicit using the user-defined material subroutine (VUMAT).

This model is consistent with the model proposed by Hurtado and Ortiz [52], where the self-energy of dislocations and the energy of dislocation steps at the boundary of the solid in the context of surface effects were accounted for in the CP theory. Horstemeyer et al. [53] performed atomistic simulations of plasticity via the embedded atom method (EAM) for single-crystal f.c.c. metals. The results of molecular-dynamics simulations indicated that plastic deformation was intrinsically inhomogeneous and the yield strength scaled inversely with the volume-to-surface area ratio, even in the absence of strain gradients. This model also complies with the EMSGCP theory. Higher-order strain-gradient models for single-crystal micro-pillars were also developed by Zhang and Aifantis [54] to describe the deformation behaviour of single-crystal micro-pillars.

Experimental data in micro-pillar deformation typically show several discrete slip bursts (Fig. 5), where the stress remains almost constant while the strain jumps discontinuously to increasing values. Zhang and Shang [55] formulated a continuum model accounting for strain bursts observed in the experiments by considering the intermittent space and time displaying in displacements of the pillar and constructing the microscopic boundary conditions in the hybrid loading mode. Zhang and Aifantis [54] used strain gradients to capture strain bursts that were experimentally observed in single-crystal Ni micro-pillars under compression. In that study, plastic deformation was realised through slip zones in a gauge region, where they were divided into elastic and plastic zones, and a strain burst occurred when two adjacent zones deformed plastically. The predicted results were in good agreement with the experimental stress-strain curves. On the other hand, Ng and Nyan [56] developed a Monte Carlo model, which could predict statistical aspects of the deformation process, including the stochastic nature of the stress-strain relation and the power-law distribution of the burst size.

FE implementations of phenomenological models such as dislocation starvation and dislocation nucleation have been employed to investigate the effect of physics on micro-pillar deformation. For instance, Greer and Nix [17] proposed a phenomenological model for sub-micron sized pillars, where initially present mobile dislocations annihilated in the vicinity of a free surface. It was given as

$$s_{starv} = 0.5 \mu b \sqrt{\rho} + 1.4 \frac{\mu b}{4\pi a(1 - \vartheta)} \ln\left(\frac{\alpha a}{b}\right), \quad (7)$$

where ϑ and α are the Poisson's ratio and constant of order unity, and where the instantaneous pillar diameter $a = a_0(1 - \varepsilon)^{0.5}$ and density $\rho = \rho_0 + \frac{(\delta - 1)}{b} \frac{\varepsilon_p}{M}$ with a_0 and ρ_0 being the pillar diameter and the initial dislocation density, ε and ε_p are the overall engineering strain and plastic strain, M is the Schmid factor and δ is the breeding coefficient, representing the inverse of the distance a dislocation travels before replicating itself. This model captured one single strain burst followed by elastic loading. Following that work, Jérusalem et al. [57] characterized starvation of pre-existing dislocations and nucleation of new ones independently by two references with $S_{0,starv}$ being initial starvation CRSS and $S_{0,nuc}$ a reference nucleation CRSSs for each slip system i as follows:

$$s_0^i = \text{Min}\left(\left(1 - \frac{\varepsilon_p}{\varepsilon_p^{starv}}\right) s_{0,starv} + \frac{\varepsilon_p}{\varepsilon_p^{starv}} s_{0,nuc}, s_{0,nuc}\right), \quad (8)$$

where

$$s_{starv} = 0.5 \mu b \sqrt{\rho_0} + 1.4 \frac{\mu b}{4\pi a_0(1 - \vartheta)} \ln\left(\frac{\alpha a_0}{b}\right).$$

In this equation, ε_p^{starv} is a model parameter corresponding to plastic strain, for which nucleation dislocation was more favourable than dislocation starvation and at

which the nucleation CRSS $\sigma_{0,\text{nuc}}$ was reached. Here, the CRSS equalled to $\sigma_{0,\text{starv}}$ at initial yielding and then linearly increased as a function of ϵ_p as the mobile dislocations moved towards the free surface, while nucleation processes became increasingly more prevalent. Once all mobile dislocations were annihilated, plasticity was fully nucleation-driven.

4 Concluding Remarks

In this chapter we have reviewed some of the critical experimental studies in the past decades that have helped to quantify and qualify some of the essential features of deformation in the small scale. The effects of extrinsic and intrinsic parameters on the overall deformation behaviour of micro- and meso-size components have paved the way for advanced numerical modelling frameworks, which attempt to capture the deformation behaviour across length scales for given boundary constraints. With an ever-increasing capacity in computational power, numerical simulations of physically reasonable component sizes under complex loading states can now be attempted. However, much work is yet to be done, especially with the regard to developing models, which are computationally efficient and physically sound.

References

1. Greer, J.R., De Hosson, J.T.M.: Plasticity in small-sized metallic systems: intrinsic versus extrinsic size effect. *Prog. Mater. Sci.* **56**(6), 654–724 (2011)
2. Stelmashenko, N., Walls, M., Brown, L., Milman, Y.V.: Microindentations on W and Mo oriented single-crystals: an STM study. *Acta Metall. Mater.* **41**(10), 2855–2865 (1993)
3. McElhaney, K., Vlassak, J., Nix, W.: Determination of indenter tip geometry and indentation contact area for depth-sensing indentation experiments. *J. Mater. Res.* **13**(05), 1300–1306 (1998)
4. Tabor, D.: *The Hardness of Metals*. Oxford University Press, Oxford, (1951)
5. Xue, Z., Huang, Y., Hwang, K., Li, M.: The influence of indenter tip radius on the micro-indentation hardness. *J. Eng. Mater. Technol.* **124**, 371 (2002)
6. Fleck, N., Hutchinson, J.: Strain gradient plasticity. *Adv. Appl. Mech.* **33**, 295–361 (1997)
7. Acharya, A.: A model of crystal plasticity based on the theory of continuously distributed dislocations. *J. Mech. Phys. Solids* **49**(4), 761–784 (2001)
8. Fressengeas, C., Taupin, V., Capolungo, L.: An elasto-plastic theory of dislocation and disclination fields. *Int. J. Solids Struct.* **48**, 3499–3509 (2011)
9. Roy, A., Acharya, A.: Finite element approximation of field dislocation mechanics. *J. Mech. Phys. Solids* **53**(1), 143–170 (2005)
10. Qu, S., Huang, Y., Pharr, G., Hwang, K.: The indentation size effect in the spherical indentation of iridium: a study via the conventional theory of mechanism-based strain gradient plasticity. *Int. J. Plast.* **22**(7), 1265–1286 (2006)
11. Swadener, J., George, E., Pharr, G.: The correlation of the indentation size effect measured with indenters of various shapes. *J. Mech. Phys. Solids* **50**(4), 681–694 (2002)

12. Demir, E., Raabe, D., Zaafarani, N., Zaefferer, S.: Investigation of the indentation size effect through the measurement of the geometrically necessary dislocations beneath small indents of different depths using EBSD tomography. *Acta Mater.* **57**(2), 559–569 (2009)
13. Abu Al-Rub, R.K., Voyiadjis, G.Z.: Analytical and experimental determination of the material intrinsic length scale of strain gradient plasticity theory from micro- and nano-indentation experiments. *Int. J. Plast.* **20**(6), 1139–1182 (2004)
14. Uchic, M.D., Dimiduk, D.M., Florando, J.N., Nix, W.D.: Sample dimensions influence strength and crystal-plasticity. *Science* **305**(5686), 986–989 (2004)
15. Uchic, M.D., Dimiduk, D.M.: A methodology to investigate size scale effects in crystalline plasticity using uniaxial compression testing. *Mater. Sci. Eng. A* **400**, 268–278 (2005)
16. Greer, J.R., Oliver, W.C., Nix, W.D.: Size dependence of mechanical properties of gold at the micron scale in the absence of strain gradients. *Acta Mater.* **53**(6), 1821–1830 (2005)
17. Greer, J.R., Nix, W.D.: Nanoscale gold pillars strengthened through dislocation starvation. *Phys. Rev. B* **73**(24), 245–410 (2006)
18. Kiener, D., Grosinger, W., Dehm, G., Pippan, R.: A further step towards an understanding of size-dependent crystal plasticity: in situ tension experiments of miniaturized single-crystal copper samples. *Acta Mater.* **56**(3), 580–592 (2008)
19. Jennings, A.T., Greer, J.R.: Tensile deformation of FIB-less single-crystalline copper pillars. *Philos. Mag.* **91** (2010)
20. Shan, Z., Mishra, R., Asif, S.A.S., Warren, O., Minor, A.: Mechanical annealing and source-limited deformation in submicrometre-diameter Ni crystals. *Nat. Mater.* **7**(2), 115–119 (2008)
21. Rabkin, E., Nam, H.S., Srolovitz, D.: Atomistic simulation of the deformation of gold nanopillars. *Acta Mater.* **55**(6), 2085–2099 (2007)
22. Rao, S.I., Dimiduk, D., Parthasarathy, T.A., Uchic, M., Tang, M., Woodward, C.: A thermal mechanisms of size-dependent crystal flow gleaned from three-dimensional discrete-dislocation simulations. *Acta Mater.* **56**(13), 3245–3259 (2008)
23. Ng, K., Ngan, A.: Deformation of micron-sized aluminium bi-crystal pillars. *Phil. Mag.* **89** (33), 3013–3026 (2009)
24. Maass, R., Van Petegem, S., Borca, C., Van Swygenhoven, H.: In situ Laue diffraction of metallic micro-pillars. *Mater. Sci. Eng. A* **524**(1), 40–45 (2009)
25. Guruprasad, P., Benzerga, A.: Size effects under homogeneous deformation of single-crystals: a discrete-dislocation analysis. *J. Mech. Phys. Solids* **56**(1), 132–156 (2008)
26. Akarapu, S., Zbib, H.M., Bahr, D.F.: Analysis of heterogeneous deformation and dislocation dynamics in single-crystal micro-pillars under compression. *Int. J. Plast.* **26**(2), 239–257 (2010)
27. Dimiduk, D., Uchic, M., Parthasarathy, T.: Size-affected single-slip behavior of pure nickel microcrystals. *Acta Mater.* **53**(15), 4065–4077 (2005)
28. Bei, H., Shim, S., Miller, M., Pharr, G., George, E.: Effects of focused ion beam milling on the nanomechanical behavior of a molybdenum-alloy single-crystal. *Appl. Phys. Lett.* **91**(11), 111915 (2007)
29. Kiener, D., Motz, C., Rester, M., Jenko, M., Dehm, G.: FIB damage of Cu and possible consequences for miniaturized mechanical tests. *Mater. Sci. Eng. A* **459**(1), 262–272 (2007)
30. Jennings, A.T., Burek, M.J., Greer, J.R.: Microstructure versus size: mechanical properties of electroplated single-crystalline Cu nanopillars. *Phys. Rev. Lett.* **104**(13), 135503 (2010)
31. Brinckmann, S., Kim, J.Y., Greer, J.R.: Fundamental differences in mechanical behavior between two types of crystals at the nanoscale. *Phys. Rev. Lett.* **100**(15), 155502 (2008)
32. Greer, J.R., Weinberger, C.R., Cai, W.: 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**(1–2), 21–25 (2008)

33. Weinberger, C.R., Cai, W.: Surface-controlled dislocation multiplication in metal micro-pillars. *Proc. Natl. Acad. Sci.* **105**(38), 14304 (2008)
34. Kim, J.Y., Greer, J.R.: Tensile and compressive behavior of gold and molybdenum single-crystals at the nano scale. *Acta Mater.* **57**(17), 5245–5253 (2009)
35. Nix, W.D.: Mechanical properties of thin films. *Metall. Mater. Trans. A* **20**(11), 2217–2245 (1989)
36. Kim, J., Jang, D., Greer, J.R.: Tensile and compressive behavior of tungsten, molybdenum, tantalum and niobium at the nanoscale. *Acta Mater.* **58**(7), 2355–2363 (2010)
37. Gao, H., Huang, Y., Nix, W., Hutchinson, J.: Mechanism-based strain gradient plasticity–I. Theory. *J. Mech. Phys. Solids* **47**(6), 1239–1263 (1999)
38. Begley, M.R., Hutchinson, J.W.: The mechanics of size-dependent indentation. *J. Mech. Phys. Solids* **46**(10), 2049–2068 (1998)
39. Nix, W.D., Gao, H.: Indentation size effects in crystalline materials: a law for strain gradient plasticity. *J. Mech. Phys. Solids* **46**(3), 411–425 (1998)
40. Yuan, H., Chen, J.: Identification of the intrinsic material length in gradient plasticity theory from micro-indentation tests. *Int. J. Solids Struct.* **38**(46), 8171–8187 (2001)
41. Huang, Y., Qu, S., Hwang, K., Li, M., Gao, H.: A conventional theory of mechanism-based strain gradient plasticity. *Int. J. Plast.* **20**(4), 753–782 (2004)
42. Wang, Y., Raabe, D., Klüber, C., Roters, F.: Orientation dependence of nanoindentation pile-up patterns and of nanoindentation microtextures in copper single-crystals. *Acta Mater.* **52**(8), 2229–2238 (2004)
43. Zaafarani, N., Raabe, D., Roters, F., Zaefferer, S.: On the origin of deformation-induced rotation patterns below nanoindents. *Acta Mater.* **56**(1), 31–42 (2008)
44. Demiral, M., Roy, A., Silberschmidt, V.V.: Indentation studies in bcc crystals with enhanced model of strain-gradient crystal plasticity. *Comput. Mater. Sci.* **79**, 896–902 (2013)
45. Demiral, M., Roy, A., El Sayed, T., Silberschmidt, V.V.: Influence of strain gradients on lattice rotation in nano-indentation experiments: a numerical study. *Mater. Sci. Eng. A* **608**, 73–81 (2014)
46. Zhang, H., Schuster, B.E., Wei, Q., Ramesh, K.T.: The design of accurate micro-compression experiments. *Scripta Mater.* **54**(2), 181–186 (2006)
47. Schuster, B.E., Wei, Q., Hufnagel, T.C., Ramesh, K.T.: Size-independent strength and deformation mode in compression of a Pd-based metallic glass. *Acta Mater.* **56**(18), 5091–5100 (2008)
48. Chen, C.Q., Pei, Y.T., De Hosson, J.T.M.: Effects of size on the mechanical response of metallic glasses investigated through in situ TEM bending and compression experiments. *Acta Mater.* **58**(1), 189–200 (2010)
49. Raabe, D., Ma, D., Roters, F.: Effects of initial orientation, sample geometry and friction on anisotropy and crystallographic orientation changes in single-crystal microcompression deformation: a crystal-plasticity finite-element study. *Acta Mater.* **55**(13), 4567–4583 (2007)
50. Shade, P.A., Wheeler, R., Choi, Y.S., Uchic, M.D., Dimiduk, D.M., Fraser, H.L.: A combined experimental and simulation study to examine lateral constraint effects on microcompression of single-slip oriented single-crystals. *Acta Mater.* **57**(15), 4580–4587 (2009)
51. Demiral, M., 2012, Enhanced gradient crystal-plasticity study of size effects in bcc metal, Ph. D. thesis, Loughborough University, UK
52. Hurtado, D.E., Ortiz, M.: Surface effects and the size-dependent hardening and strengthening of nickel micro-pillars. *J. Mech. Phys. Solids* **60**(8), 1432–1446 (2012)
53. Horstemeyer, M., Baskes, M., Plimpton, S.: Length scale and time scale effects on the plastic flow of fcc metals. *Acta Mater.* **49**(20), 4363–4374 (2001)
54. Zhang, X., Aifantis, K.: Interpreting strain bursts and size effects in micro-pillars using gradient plasticity. *Mater. Sci. Eng. A* **528**, 5036–5043 (2011)

55. Zhang, X., Shang, F.: A continuum model for intermittent deformation of single crystal micropillars. *Int. J. Solids Struct.* **51**(10), 1859–1871 (2014)
56. Ng, K., Ngan, A.: Breakdown of Schmid's law in micro-pillars. *Scripta Mater.* **59**(7), 796–799 (2008)
57. Jérusalem, A., Fernández, A., Kunz, A., Greer, J.R.: Continuum modeling of dislocation starvation and subsequent nucleation in nano-pillar-compressions. *Scripta Mater.* **66**(2), 93–96 (2012)

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