Size effects in miniaturized polycrystalline FCC samples: Strengthening versus weakening

M.G.D. Geers a,*, W.A.M. Brekelmans a, P.J.M. Janssen b,a

a Eindhoven University of Technology, Department of Mechanical Engineering, Materials Technology, P.O. Box 513, 5600 MB Eindhoven, The Netherlands
b Netherlands Institute for Metals Research (NIMR), P.O. Box 5008, 2600 GA Delft, The Netherlands

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Abstract

This paper focuses on a computational-experimental analysis of sample geometry dominated and grain dominated size effects in miniaturized polycrystalline FCC components, where the grain size, orientation and grain boundaries play an important role. Experimental and numerical findings elucidate the joint contribution of first-order and second-order size effects for this type of components. The intrinsic competition between the weakening and strengthening contributions resulting from these effects is commented and further analysed. It is shown that a second-order crystal plasticity model is needed to account for the simultaneous contributions of both size effects.

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1. Introduction

The past decade has been marked by a substantial progress in the proper understanding and modelling of the mechanics of materials at small length scales. The ongoing miniaturization that has driven the technological developments in micro-electronics and micro-electromechanical systems (MEMS), is one of the main industrial evolutions that is facing the intriguing mechanical problems that play a role at smaller scales. Upon miniaturization, the size of microstructures is generally no longer negligible with respect to the component size as used in many microsystems and sub-micron applications. Microstructural effects, macroscopically triggered gradient effects and surface effects jointly appear and constitute the various size effects that can be observed. Classical continuum mechanics theories fail to describe these phenomena and higher-order micromechanical concepts are required to obtain an appropriate prediction of the mechanical behaviour of miniaturized structures.
From an experimental perspective, most attention on size effects in miniaturization has been given to strengthening effects in metals, whereby micro-bending experiments (Stölken and Evans, 1998; Takahiro et al., 2000), nano-indentation experiments (Ma and Clarke, 1994; McElhaney et al., 1998; Nix and Gao, 1998; Elmustafa et al., 2000; Tymiak et al., 2001; Shan and Sitaraman, 2003; Zhao et al., 2003), torsion experiments (Fleck et al., 1994; Fleck and Hutchinson, 1997) or thin film analyses (Haque and Saif, 2003; Greer et al., 2005; Zong and Soboyejo, 2005) were the main set-ups used. In almost all cases, a pronounced strengthening has been found at decreasing specimen scale. Less attention was given to the intrinsic role of the microstructure in each of these experimental set-ups, whereby the precise granular structure (grain size, grain orientations and prior deformation history, i.e. initial dislocation densities and lattice distortions) was not considered as an important influencing factor. A comparable grain size was mostly accepted as a sufficient condition for analysing the occurring size effect. If the number of grains gets small, clear microstructural effects will contribute and all of these effects cannot be separated trivially in the experimental results. Only few papers seem to deal with this microstructural contribution, and mostly in a different context (Miyazaki et al., 1978, 1979; Kals and Eckstein, 2000; Nakamachi et al., 2000; Raulea et al., 2001; Klein et al., 2001; Hansen, 2005). The present paper concentrates on this important aspect, since it will be shown that depending on the specimen size and the underlying microstructural geometry, weakening effects may occur jointly with strengthening effects.

From a computational point of view, several higher-order constitutive models (Li and Cescotto, 1996; de Borst and Pamin, 1996; Fleck et al., 1994; Fleck and Hutchinson, 1997; Shu and Fleck, 1999; Chen and Wang, 2000; Forest et al., 2000; Fleck and Hutchinson, 2001; Gao and Huang, 2001; Bazant and Guo, 2002; Gao and Huang, 2003; Niordson and Hutchinson, 2003; Qiu et al., 2003; Mughrabi, 2004; Creighton et al., 2004; Gudmundson, 2004; Lee et al., 2005; Gurtin and Anand, 2005) have been proposed to capture the experimentally observed strengthening effects. Most of these models are continuum type models, in which the individual grains of the underlying metal microstructure are not explicitly taken into account. More recently, modelling efforts have been made to predict strain gradient effects at the crystalline level, where a number of advanced strain gradient crystal plasticity models have resulted (Bronkhorst et al., 1992; Forest, 1998; Ashmawi and Zikry, 2000; Acharya, 2001; Mughrabi, 2001; Evers et al., 2002; Arsenlis and Parks, 2002; Gurtin, 2002; Evers et al., 2004a,b; Cheong et al., 2005; Han et al., 2005a,b; Yefimov and van der Giessen, 2005a; Nicola et al., 2005a; Yefimov and van der Giessen, 2005b) and approaches related to e.g. discrete dislocation plasticity (Shu et al., 2001; Kubin and Mortsensen, 2003; Zaiser and Aifantis, 2003; Deshpande et al., 2003; Nicola et al., 2005b). The model of Evers et al. (2004b) will be used for further analysis here, since it incorporates slip gradient strengthening, a detailed grain boundary treatment and special external boundary conditions into the formulation.

After a general classification of size effects, this paper will focus on a computational-experimental analysis of sample geometry dominated and grain dominated size effects in miniaturized polycrystalline FCC samples, where the grain size, orientation and grain boundaries play an important role. The experimental analysis focuses on the free surface effect that emerges if the grain size is no longer negligible with respect to the sample dimensions. A series of tensile experiments on pure aluminum samples has been carried out, whereby careful material and specimen preparation allowed to rule out higher-order size effects and grain statistics effects. A pronounced grain size dependence is thereby observed, showing a first-order weakening effect if the ratio between the sample thickness and the grain size reduces. Since conventional continuum gradient plasticity models do not account for the granular nature of the microstructure, they are unable to reveal the first-order weakening effects mentioned above. Therefore, a recently developed strain gradient dependent crystal plasticity approach will be adopted, which incorporates the first-order influence and a second-order contribution through its intrinsic scale dependence. The heterogeneous deformation-induced evolution and distribution of geometrically necessary dislocations (GNDs) are incorporated into a phenomenological continuum theory of crystal plasticity, of which the theoretical and numerical details have been published recently (Evers et al., 2004a,b). In the present paper, this framework is applied to FCC multi-crystalline samples in tension and bending, where the competitive influence of first- and second-order size effects is the main issue addressed.

2. An engineering classification of size effects

In order to interpret the occurrence of size effects from the right perspective, it is recommended to make a rough classification of known size effects. Important to emphasize, is the fact that there is no universal trend...
reflecting ‘smaller is stronger’. It all depends on the type of effect that is being considered, whereas all effects contribute in the engineering practice or parallel experimental analyses.

2.1. Microstructural or intrinsic size effects

The oldest and best-known size effects in polycrystalline metals are certainly those that have been used by metallurgists throughout the years to optimize the performance of several alloys. This category will further be denoted as ‘microstructural size effects’, reflecting the intrinsic role of the dimensions of phases and particles in the microstructure of a heterogeneous or multi-phase metal. From a materials science perspective, these effects originate from the elementary physical processes producing deformation on the sub-micron and nano-scale level. This category is also known as ‘intrinsic’ size effects (Sevillano et al., 2001). Typical examples are the Hall–Petch effect (reflecting the grain size dependence of the hardening of metals), the Friedel effect and the Orowan effect. Microstructural size effects are the result of interacting unit deformation processes in all phases, in which many microstructural length scales play an important role (Burgers vector length, grain size, obstacle size, obstacle spacing, grain boundary width, etc.). These effects may both lead to strengthening (e.g. Hall–Petch) or weakening (e.g. inverse Hall–Petch, Chokshi et al. (1989), Arzt (1998)), ineffective blocking of slip by second phase particles (Benzerga et al., 2001) upon decreasing length scales.

2.2. Statistical size effects

A special case of size effects is found in the polycrystalline nature of the metal itself. For small geometrical dimensions, the number of grains in one of the spatial directions may become small, triggering an orientation dependence and additionally a grain boundary dependence. This category of size effects becomes apparent for specific ratios of the component size in relation to the characteristic microstructural size. This interesting case will be discussed in more detail in Section 3 on first-order grain size effects.

2.3. Lattice curvature and strain gradients

A widely discussed size effect that explains the strengthening of many metals at small length scales, is the lattice curvature effect. This category of size effects is commonly called ‘gradient effects’. Keeping all geometrical proportions identical, downscaling a component will naturally lead to an increase of the gradients of strain. These gradients have to be accommodated geometrically in the lattice by curvature, which is intrinsically limited through the lattice structure. The only physical manner to accommodate the curvature is by the introduction of extra dislocations in the lattice, commonly denoted as geometrically necessary dislocations (GNDs). The most important characteristic of these dislocations is their geometrical connection to the curvature, whereby e.g. the orientation of extra atomic half planes is geometrically determined for edge GNDs. Like any other dislocation, GNDs obstruct plastic slip, contributing to additional hardening. However, due to their geometrical orientation, they may generate long-range back-stresses in the lattice, possibly resulting in a pronounced effect on the mechanical response. An often cited illustration of gradient effects was given by Stölken and Evans (1998), who performed micro-bending of thin annealed nickel foil. Experimental results, showing the relevance and the importance of this (strengthening) size effect, can be found throughout the literature, e.g. torsion of thin copper wires (Fleck et al., 1994), micro- and nano-indentation (Ma and Clarke, 1994; McElhaney et al., 1998; Elmustafa et al., 2000), nano-scale bending on single crystal silicon (Takahiro et al., 2000), bending of copper layers in printed circuit boards (Geers et al., 2005), etc.. In almost all cases, significant effects were measured.

2.4. Surface or interfacial constraints

A special category of size effects are due to the interaction of the carriers of plastic slip with the external boundary or with interfaces with a different material. From a physical point of view, similar ‘unit processes’ as those identified for microstructural size effects are playing a role here. From the engineering perspective, these size effects appear as a different category, since the external boundary conditions and material interfaces can be influenced during or after the processing of the material. In the case of small, thin or multi-layered
structures, all material is relatively close to a physical boundary or interface. These size effects therefore naturally induce a dimensional constraint, as categorized by Arzt (1998). The particular physical condition at the boundary or interface may either restrict (constrained case) or freely facilitate (unconstrained case) the deformation nearby the boundary. For polycrystalline metals, plastic slip is carried by dislocations which can either be blocked at the boundary (constrained) or glide out of it (unconstrained), leaving behind a surface step. Hard coatings or oxide layers are typical examples that may obstruct plastic slip up to a certain level. The influence of the boundary layer generally covers a certain volume, which explains the importance of the surface-to-volume ratio as found in experimental analyses (Kals and Eckstein, 2000). Surface or interfacial constraints may induce strengthening or weakening, depending on the physical boundary conditions and the deformation mechanisms that are being influenced. For metallic systems, hard boundary layers generally lead to strengthening upon downscaling of a component. Unconstrained or ‘free’ boundaries generally contribute in the opposite sense.

3. First-order grain size effects

Another interesting way to assess size effects, is by distinguishing first-order effects from second-order effects. First-order effects cover all effects resulting from the discrete granular anisotropic nature of the microstructure, however excluding contributions due to gradients of deformation (strain gradients, slip gradients, etc.). Obviously, lattice curvature effects and boundary effects are clearly second-order effects. From a mathematical point of view, first-order effects can be modelled by ‘standard’ microstructural theories that rely on the principle of local action. Conventional crystal plasticity is a typical relevant example used in a first-order analysis of polycrystalline metals. Microstructural size effects may be partially of a first-order and partially of a second-order nature. Grain boundaries well illustrate this. The continuum influence of the orientation mismatch between two neighbouring grains is well addressed in a conventional crystal plasticity framework. The additional grain boundary hardening, that may result from the pile-up of dislocations at a grain boundary is a typical second-order effect. In this section, a clear illustration of statistical size effects will be given, where a non-negligible first-order contribution will be emphasized. The quantitative relevance of these effects puts a serious constraint on the model to be used to address both effects, i.e. first-and second-order.

In order to assess grain statistical size effects, specimens are considered in which the number of grains across the specimen dimensions is small. The relevance of this analysis is illustrated in Fig. 1, where a cross-section of a part of a free-standing metallic MEMS-component (aluminum-based RF-MEMS) is depicted. This figure shows that the presence of a limited number of grains (here in the width direction) or even a single grain (here in the thickness direction) is a practical and relevant issue for the engineering community.

3.1. Grain statistical effects, a numerical assessment

The numerical analysis of the first-order contribution of grain statistics is conducted on a polycrystalline sample with a limited number of grains. To this purpose, a local, standard crystal plasticity model has been used (Asaro and Rice, 1977; Peirce et al., 1982; Kalidindi et al., 1992; Bronkhorst et al., 1992). The governing equations are briefly summarized below:

- The multiplicative decomposition of the deformation gradient tensor $\mathbf{F}$ into its elastic part $\mathbf{F}_e$ and its plastic part $\mathbf{F}_p$

  $$\mathbf{F} = \mathbf{F}_e \cdot \mathbf{F}_p$$  

(1)

- The elasticity equation, coupling the second Piola–Kirchhoff stress tensor $\mathbf{S}$ to the elastic Green–Lagrange strain $\mathbf{E}_e$ through the FCC-anisotropic elastic constitutive tensor $^{\text{\text{4}}} \mathbf{C}$

  $$\mathbf{S} = ^{\text{\text{4}}} \mathbf{C} : \mathbf{E}_e, \text{ with } \mathbf{E}_e = \frac{1}{2} (\mathbf{F}_e^T \cdot \mathbf{F}_e - \mathbf{I})$$

(2)

- The crystallographic split, coupling the plastic velocity gradient tensor $\mathbf{L}_p$ to the crystallographic slip rates $\mathbf{\dot{\gamma}}^s$ and the Schmid tensor $\mathbf{P}^s_\alpha = \mathbf{\overline{m}_0} \mathbf{\overline{n}_0}$, with $\mathbf{\overline{m}_0}$ and $\mathbf{\overline{n}_0}$ the slip direction and slip plane normal, respectively
\[ F_p = L_p \cdot F_p, \text{ with } L_p = \sum_x \dot{\gamma}^x P_0^x \]

- The flow rules for the crystallographic slip, involving the resolved shear stress \( \tau^x = S : P_0^x \), the reference slip rate \( \dot{\gamma}_0 \) on each slip system \( x \) and the slip resistance \( s^x \)

\[ \dot{\gamma}^x = \dot{\gamma}_0 \left| \frac{\tau^x}{s^x} \right| \operatorname{sgn}(\tau^x) \]

- The slip system hardening, expressed in terms of the slip resistance, in which the moduli \( h^{x\beta} \) reflect the self-hardening on slip system \( x (\beta = x) \) and the latent hardening on slip system \( x \) due to cross-slip on system \( \beta (\beta \neq x) \)

\[ s^x = \sum_\beta h^{x\beta} |\dot{\gamma}^\beta| \]

- A Kocks-type hardening equation, giving the moduli \( h^{x\beta} \), in which \( h_0, s_a \) and \( a \) are material self-hardening parameters and in which \( q^{x\beta} \) represents the latent hardening parameter

\[ h^{x\beta} = h_0 \left( 1 - \frac{s^a}{s_k} \right)^a q^{x\beta} \]

The solution of these equations in the context of a finite element framework follows standard procedures, which can be found in many contributions in the literature (Bronkhorst et al., 1992; Evers et al., 2002).

For the analysis of first-order statistical size effects, an aluminum reference specimen with length \( l = 10 \) mm, width \( w = 4 \) mm and thickness \( t = 0.5 \) mm is considered. The microstructure has a typical mean grain size of 100 \( \mu \)m, resulting in a limited number of grains across the thickness (5 for the reference specimen). Note, that second-order effects are hardly expected for these dimensions, whereas the resulting first-order effects apply to all scales. The grain structure in the specimen has been generated using a Voronoi tessellation, which has been shown to provide realistic approximates of the real grain structure of metallic materials (Wu and Guo, 2000).

Two downscaled specimens have been considered as well, whereby the microstructural size and orientations (distributed randomly) are kept constant. The smallest specimen is 100 \( \mu \)m thick, the medium-sized one 250 \( \mu \)m thick. All other specimen dimensions are proportionally scaled. Over 2000 3D cubic elements have been used to discretize the microstructures of all specimens. For each specimen geometry, five samples have been considered, taken from the same random orientation distribution space. The material is aluminum,
for which the material parameters, as used in the set of crystal plasticity equations given above, have been adopted from Kumar and Dawson (2000).

3.1.1. The tensile case

The mechanical response obtained from the tensile test simulations performed on the specimens described above are shown in Fig. 2. Two trends are clearly observable:

1. The mean flow stress and hardening rate decrease for thinner specimens, whereby the microstructure is kept constant. Obviously, this effect indicates ‘smaller is weaker’.
2. The spread around the mean response increases for thinner specimens. Clearly, individual grains tend to have a larger influence on the overall mechanical behaviour.

The weakening trend found is a typical first-order effect, that ensues from the gradual loss of polycrystallinity. Indeed, if the microstructural dimensions are kept constant, the reduction of the specimen dimensions will always trigger an increased influence of individual grains if a limited number of grains across at least one of the specimen directions is left. Note that this effect depends on the fact that surface grains are less obstructed and hence more deformable. Essentially, uniformity is lost due to the microstructural nature of the specimen. The increased spread is mainly due to the mutual differences between the grains composing the specimen, i.e. their orientation. Manufacturing samples with a pronounced texture or even a nearly single orientation would reduce this weakening effect (depending on the orientation) and the increased uncertainty considerably.

Note that the results found here, are consistent with the experimental results from Kals (1998).

3.1.2. The bending case

The first-order weakening effect becomes logically more pronounced for macroscopically non-homogeneous loading cases. This is the case for bending, where a clear variation of the strains over the cross-sections is present. Consequently, the dependence on the orientation of even fewer grains becomes manifest, as shown in the mean curves depicted in Fig. 3. Again, the trend is ‘smaller is weaker’ and the effect is more pronounced. ‘Weakly’ oriented grains in the sample have a larger influence in bending compared to the tensile case. Again the effect is due to the loss of polycrystallinity and the accompanying increased influence of surface grains.

3.2. Grain size effects, experimental analysis

Experimental evidence for first-order size effects can e.g. be found in Miyazaki et al. (1978, 1979), Kals (1998), Kals and Eckstein (2000), Nakamachi et al. (2000), Janssen et al. (submitted for publication).
the present paper, particular attention is focused on the interpretation of the results found in Janssen et al. (submitted for publication). In this work, a careful experimental analysis has been conducted in order to identify the grain size influence at a gradually reducing number of grains across the thickness in a tensile specimen. The specimens were carefully prepared, in order to minimize secondary influences from processing or texture, see Janssen et al. (submitted for publication) for full details on the experimental procedures. The results found in that work are schematically represented in Fig. 4. This figure qualitatively depicts the true stress for a given strain as a function of $\lambda_s$, which is the ratio of the specimen thickness $t$ to the grain size $d_s$ as measured on the specimen surface. The grain size measured through the thickness of the specimen is denoted by $d_t$. The smallest thickness considered equalled 95 $\mu$m, from which it can be safely concluded that all effects observed are most dominantly of the first-order type. As qualitatively visible in Fig. 4, the following conclusions can be drawn:

- Upon miniaturization, there is no unique manner to draw generalized conclusions on the grain size, since they might be different along the different specimen axes.
- Keeping the dimensions of the grains constant (and hence also $d_t$), naturally leads to a drop in the true stress upon reduction of the specimen thickness. This is a clear statistical grain size effect.

![Fig. 3. Normalized bending force versus normalized displacement in the bending of thin sheets.](image)

![Fig. 4. Qualitatively found grain size dependence for tensile sheets with a limited number of grains across the thickness.](image)
• The classical Hall–Petch effect, which reflects an increased hardening upon grain refinement, no longer applies if the number of grains across the thickness approaches unity.
• For specimens with a single grain across the thickness and a number of grains across the width, there seems to be a negligible dependence on the grain size measured on the surface. Further work is in progress to refine the conclusions in this regime.

Again, weakening is found to be the overall first-order effect present upon size reduction. The main explanation for the effect is the absence of internal ‘mid-plane’ grain boundaries for specimens with a single grain across the thickness. Specimens with a columnar structure (either pancake or needle-like) as typically shown in Fig. 1, are nearly having through thickness grain boundaries only. It is obvious from this analysis that care must be taken in formulating trends on size effects independently from the size of the underlying microstructure.

4. Second-order effects

Considering the results shown in the previous section, and the non-negligible contribution of first-order effects, a microstructural model is needed for the proper analysis of second-order effects present in MEMS-structures as shown in Fig. 1. Engineering problems for which the first-order effects are intrinsically present (prior to second-order effects) cannot be analyzed with continuum models that ‘ignore’ the discreteness (i.e. the granular nature) of the microstructure, e.g. continuum strain gradient plasticity models (Aifantis, 1984; Fleck et al., 1994; Xia and Hutchinson, 1996; Li and Cescotto, 1996; de Borst and Pamin, 1996; Fleck and Hutchinson, 1997; Haung et al., 1997; Gao et al., 1999; Chen and Wang, 2000; Gao and Huang, 2001; Fleck and Hutchinson, 2001; Bažant and Guo, 2002). However, note that if the number of grains is sufficiently large compared to the specimen dimensions these continuum theories may remain well applicable.

The analysis of second-order effects is further assessed from a numerical perspective, using a strain gradient crystal plasticity model. The presence of grains, the treatment of grain boundaries and the incorporation of long range stress fields that accompany GNDs are thereby key ingredients. The model which will be used to this purpose has been elaborated in detail in Evers et al. (2004a) and various aspects on its use for multi-crystalline FCC samples with 12 slip systems, including special grain boundary conditions, have been highlighted in Evers et al. (2004b). Attention is therefore restricted here to a summary of the main features of this model. The outline of the model is presented in Fig. 5. Main characteristics of this strain gradient crystal plasticity framework and its numerical implementation are:

• The total deformation is decomposed in an elastic part and a plastic part using the classical multiplicative split. A Hookean constitutive model is used to compute the stress tensor from the elastic Green–Lagrange strain measure.

![Fig. 5. Dislocation density based strain gradient crystal plasticity framework.](image-url)
• The decomposition of the plastic velocity gradient tensor into the contributions of the 12 FCC slip systems constitutes the classical crystallographic split.
• A viscoplastic slip law has been adopted for the evolution of the slip rates on each of the slip planes, in which the effective resolved shear stress $\tau_{\text{eff}}$ and a slip resistance $s^a$ enter the evolution equations.
• The GND densities are directly computed from the gradient of the crystallographic slips, whereby the obtained sign is representative for the polarity of the GNDs.
• The effective stress is the difference between the classical resolved shear stress $\tau^a$ and the resolved back-stress $\tau_b$. The back-stress tensor is computed locally from a circular patch in which the stress contributions from all GNDs present have been analytically integrated.
• The slip resistance $s^a$ incorporates the short range interactions between SSDs (statistically stored dislocations) and GNDs, for which the work of Franciosi and Zaoui (1982) was used to quantify the resistances $s^a$.
• The SSD densities are described by a generalized form of Essmann and Mughrabi (1979), in which the dislocation accumulation is governed by the average dislocation segment length and in which the dislocation annihilation is controlled by a critical annihilation length.
• Grain boundaries are incorporated by: (1) assigning an initial GND density to each internal grain boundary, which is computed from the (grain boundary projected) orientation mismatch between neighbouring grains; (2) by constraining the net normal slip at each grain boundary, whereby the grain boundaries are modelled as impenetrable. Note that Evers et al. (2004b) has shown that the initial GND density provides a grain size dependence of the initial flow stress, which is an essential characteristic of the Hall–Petch behaviour. The geometrical model for the grain boundary is an adequate representation of a low-angle boundary but evidently debatable for high-angle boundaries.
• The resulting boundary value problem consists of the standard equilibrium equation and a second equation which enforces the geometrical relation between the nodal GNDs (acting as degrees-of-freedom) and the crystallographic slips in the weak sense.

All analyses presented in the next subsections have been performed on FCC copper, for which all required material parameters are given in Evers et al. (2004b). Note that the applied strain gradient crystal plasticity model naturally incorporates a back stress that contributes to kinematic hardening effects. The influence on the unloading behaviour is thereby an important issue, which is illustrated in Bayley et al. (in press).

4.1. The tensile case

The tensile case was examined in Evers et al. (2004b) for a sample of 12 grains with a fixed initial orientation of the individual grains. In the present research, four samples will be investigated, consisting of 1, 4, 12 and 50 grains, respectively. This permits to acquire a better understanding of the role of the number of grains across the specimen dimensions in assessing the combined presence of first-order and second-order effects in the mechanical response. The external geometry of the sample and the displacement boundary conditions are identical to those defined in Evers et al. (2004b), i.e. a plane-stress rectangular sample of length $L$, height $H = 0.5L$ and thickness $T = 0.1L$. The discretizations used for the different number of grains are depicted in Fig. 6. All orientations have been taken randomly, whereby the single grain case is near-cube. Evidently, depending on the number of grains in the sample, the individual orientations will have a quantitative influence on the response. Attention will therefore be restricted to the qualitative differences between the samples, whereby the exact values may change upon altering the individual orientation of grains.

The first analysis is carried out on the basis of the same sample as used by Evers et al. (2004b), i.e. with 12 grains only. In here, different orientation sets have been considered, all originating from a random orientation distribution. The first-order influence of the individual orientations of the grains can then be compared to the second-order effect reported in Evers et al. (2004b). The total sample length ($L$) used for this analysis equalled 1 mm. The resulting orientation influence is depicted in Fig. 7, where the orientation set ‘0’ indicates the set that has been used by Evers et al. (2004b). Clearly, the results presented by Evers et al. (2004b) happened to coincidently correspond with the weakest response of all orientation sets sampled. The results essentially show that for samples with a limited number of grains across the dimensions, a first-order influence of the
grain orientations comes into play, which is of the same order of magnitude as the strengthening effect, at least for the considered sample size and deformation range. Evidently, the smaller the samples, the more pronounced the second-order hardening effect will be. Note that the typical scatter induced by local grain orientations has also been reported in the literature for thin-film specimens with very narrow gauge sections, Espinosa et al. (2003, 2004). A quantitative assessment of the scatter is only possible if detailed information on the texture or orientation distribution in the samples is available.

Next, the grain size dependence is analyzed. The average grain diameter used is computed by approximating the grain areas with circles of equal surface. The dimensions of the samples are scaled in order to obtain a nearly equal grain size for the different multi-grain samples. For these analyses, ‘free’ external boundaries were used, i.e. the slip is not constrained externally, but at internal grain boundaries only. The axial tensile stress obtained at a deformation of 1% is shown in Fig. 8 as a function of the grain size.

Fig. 6. Discretized tensile samples with a different number of grains. (a) 1 grain (b) 4 grains (c) 12 grains and (d) 50 grains.

Fig. 7. Influence of the grain orientation sampling on the tensile response of a 1 mm FCC tensile specimen; orientation ‘0’ is taken from Evers et al. (2004b).
The following observations can be made:

1. The size dependence is fading away for large grain sizes. Nevertheless, the sample with the largest number of grains gives the largest tensile stress. This is a clear first-order effect.

2. For small grain sizes (and consequently small sample sizes), a pronounced hardening effect occurs, which is here due to first-order and second-order effects. The first-order effect generally leads to a weaker response for samples with a limited number of grains across the thickness. The second-order effect depends strongly on the grain size, since plastic slip is obstructed through the grain boundaries. The strengthening observed is clearly due to grain boundary hardening on the one hand and the difference between the number of grains across the width of the sample on the other hand.

3. For intermediate grain sizes (40–100 \( \mu \text{m} \)), first- and second-order effects are quantitatively of the same order and their interaction increases the dependence on the individual orientations of grains in the weakest cross-section of the sample. This orientation dependence was also emphasized previously for the particular case of the 12-grain sample.

4. Note that for the considered (realistic) samples with through-thickness grains, the precise definition of the grain size in the 3D sense is of course ill-posed. Grains may be either pancake-like or needle-like. A log–log plot in Fig. 8 with an inverse grain size on the horizontal axis (suggesting a Hall–Petch trend) may therefore be misleading. The results essentially indicate that both thickness and grain size interact in delivering the mechanical response. The separate influence of the thickness (for constant grain distributions in the plane of the sample) may be addressed separately (see also Evers et al. (2004b)), but this requires a 3D analysis if the 3D boundary effects are to be incorporated properly.

The third aspect of interest for the tensile case, is the influence of constraints at the external boundary, whereby plastic slip normal to that boundary is obstructed. Like for the case with unconstrained boundaries, strengthening results from an increase of the number of grains, which is therefore not examined again. It is more interesting here to focus on the relative strengthening of each of the constrained samples compared to the same sample with free boundaries. This strengthening \( \sigma^* \) (which is the ratio of the tensile stresses in the constrained case relative to the unconstrained case) is shown in Fig. 9. The largest relative effect is obtained for the single crystal sample, which is more pronounced for smaller grain sizes. Not surprisingly, the effect diminishes if the number of grains increases. This clearly illustrates that the second-order effect introduced through the external boundary restriction has an influence on the mechanical response, which is dependent on the grain size. This correlation cannot be predicted by continuum strain gradient plasticity models that ignore the discrete granular character of the microstructure. Again, this example illustrates in a convincing
manner that first- and second-order effects strongly interact with each other in the final mechanical response. Note, that since all results were systematically visualized as a function of the grain size, only a single curve would be found if the Hall–Petch relation would be valid for the limited amount of grains considered here. Clearly, this is no longer the case.

Fig. 10 presents the distribution of the GNDs (i.e. scalar norm of all crystallographic GND densities) for the 50-grain sample for the unconstrained and constrained case, where the mean grain size equals 8 μm and the overall strain level is 1%. This figure emphasizes that the strengthening gained from the external boundary constraint is limited to the outer layer of grains close to the external boundary. Within the sample, there is no noticeable difference between the values of the scalar GND density. The pronounced strengthening effects found for the smallest grain size in this numerical assessment have not been confirmed experimentally in the same quantitative manner. This is largely due to the severe slip constraint adopted in the grain boundary model, which has a clear quantitative influence on these results. If partial slip through a grain boundary were possible, the difference between the various samples would be obviously smaller and the strengthening much less pronounced. Nevertheless, the conclusions drawn here remain valid in a qualitative sense.

4.2. The bending case

To conclude the analysis of second-order effects, some preliminary examples of bending for the 4-grain sample and the 50-grain sample will be shown. To this purpose, the same geometry \((L \times H \times T)\) as used for the tensile tests will be adopted, whereas the boundary conditions are defined on the basis of Fig. 11. Bending is here simulated by rigorously prescribing the displacements at the edges A–B and C–D in order to accommodate a pure rotation of the boundaries towards the positions A′–B′ and C′–D′. Evidently, several alternative boundary conditions can be proposed as well. In here, attention is focused on the qualitative comparison of the mechanical response for different samples sizes and the two grain configurations. For the analysis of the results, a scaled bending moment \(M^*\) and a scaled curvature \(\kappa^*\) are used, as given by

\[
\begin{align*}
M^* &= M \times \text{constant} \\
\kappa^* &= \kappa \times \text{constant}
\end{align*}
\]

Fig. 10. Relative strengthening of multi-grain samples due to external no-slip boundary conditions.
For the adopted boundary conditions and a scaled curvature $\kappa^* = 0.04$, relative scaled bending moments are depicted as a function of the specimen length (where all other dimensions are scaled proportionally) in Fig. 12. Both cases (4 grains and 50 grains) yield the same qualitative results. Smaller specimen dimensions naturally induce strengthening. Furthermore, constraining slip at the external boundaries has an additional strengthening effect, which is more pronounced for the sample with 4 grains only. This is again consistent with the tensile test analyses, which indicated that the sensitivity to the external boundary conditions increases for a smaller number of grains. Strengthening ratios are still large compared to experimentally measured values, but again the stiff grain boundaries presumably have an excessive contribution to this.

More interesting in the context of first-order effects is the comparison between the response of the two sample types as a function of the average grain size. This dependence is shown in Fig. 13. This graph essentially shows that for large grain sizes (and sample sizes) the 4-grain sample is systematically stronger than the 50-grain sample. This is obviously due to the orientations of the grains which triggers a first-order effect here. Note that this result does not comply with a classical Hall–Petch relation, which is due to the loss of polycrystallinity. The tendency partially changes for smaller grain diameters, where the 4-grain sample is the weakest one for free boundaries, yet still the strongest one for fully constrained slip at the boundaries. It is believed that this is a clear illustration of the fact that first- and second-order effects interact if one considers samples with a limited number of grains. Note, that the quantitative differences between the various samples also depend on the local orientations of the grains (first-order effect). Depending on the texture of the sample
(or the spread in orientation of samples with a few grains only) the expected spread in Fig. 13 may be relatively small or large. Finally, the distribution of the equivalent scalar measures for the dislocation densities are depicted in Fig. 14 for the GNDs and in Fig. 15 for the SSDs, for an average grain size of 80 μm. These figures highlight that the macroscopic gradients in the deformation field tend to induce GNDs throughout the entire specimen away from the neutral axis. The no-slip constraint at the upper and lower boundary has a limited influence, which is here governed by the grain size of the boundary grains. As expected, there is no difference noticeable between the SSD densities for the slip free and no-slip boundary condition.

![Fig. 13. Scaled size dependence in bending.](image1)

![Fig. 14. GND distribution for the 50-grain bending sample with slip-free external boundaries (left) and slip-constrained external boundaries (right) [m⁻²].](image2)

![Fig. 15. SSD distribution for the 50-grain bending sample with slip-free external boundaries (left) and slip-constrained external boundaries (right) [m⁻²].](image3)
5. Conclusions

This paper focused on the qualitative existence and interaction of first-order and second-order size effects, which are expected to occur in all miniaturized metallic structures in which there are only a limited number of grains left across one of the dimensions. This is typically the case for metallic MEMS structures with feature sizes ranging from 1 µm and less to a few 100 µm.

The nature of first-order size effects has been explained and demonstrated both numerically and experimentally. It has been shown that the individual orientation of grains plays an essential role if the dimension of a structure is downscaled while keeping the microstructure 'constant'. For a random orientation of grains, downscaling the dimensions generally implies weakening, which is more pronounced for macroscopically non-homogeneous deformations (e.g. bending). The experimental analysis also indicated that the effect evolves discretely with the microstructure, where the dependence of the mechanical response on the grain size may change if e.g. there is only one grain left in one of the sample directions. It is expected that these inverse size effects can be better controlled or even minimized by carefully controlling the texture or even individual orientations of the composing grains during processing. One of the important implications is that these type of effects cannot be captured by continuum based theories that ignore the discrete character of a granular microstructure. Practical applications for which a limited number of grains come into play require an appropriate framework, i.e. a crystal plasticity approach as used here.

The parallel existence of first-order and second-order effects also makes it difficult to use continuum strain gradient plasticity models (which ignore the discreteness of individual grains) to predict the strengthening contribution expected from second-order effects. Both effects will always co-exist in the engineering practice. Second-order effects have been investigated here on the basis of a recently developed strain gradient crystal plasticity model, which intrinsically incorporates first- and second-order effects. Three types of second-order effects have been taken into account in the analysis: internal grain boundaries obstructing crystallographic slip; externally constrained boundaries with restricted slip; macroscopic gradients as present in bending tests. By means of the simulation of a tensile test and a bending test, it has been shown that second-order effects indeed trigger strengthening, but that these effects may be damped considerably by counteracting first-order effects that heavily depend on the number of grains present in the sample and their individual orientations. Furthermore, both effects may also interact, which complicates clear comparisons between sample sizes if the exact microstructure is not known in detail. In any case, the analysis has shown the superiority of a strain gradient crystal plasticity model over classical crystal plasticity models and conventional continuum strain gradient models, which constitutes a corner stone for future developments.

As a result, it is believed that the engineering impact of all the occurring effects is reasonably well understood. Precise quantitative predictions and experimental results are still missing, since adequate data on the microstructure needs to be measured and incorporated, whereby improved grain boundary models need to be developed to relax the stiff no-slip condition used here. This is the aim of future work, where metallic MEMS structures will be used as a subject for numerical-experimental investigations.

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