Modeling the dependence of strength on grain sizes in nanocrystalline materials

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Abstract
A model was developed to describe the grain size dependence of hardness (or strength) in nanocrystalline materials by combining the Hall–Petch relationship for larger grains with a coherent polycrystal model for nanoscale grains and introducing a log-normal distribution of grain sizes. The transition from the Hall–Petch relationship to the coherent polycrystal mechanism was shown to be a gradual process. The hardness in the nanoscale regime was observed to increase with decreasing grain boundary affected zone (or effective grain boundary thickness, $\Delta$) in the form of $\Delta^{-1/2}$. The critical grain size increased linearly with increasing $\Delta$. The variation of the calculated hardness value with the grain size was observed to be in agreement with the experimental data reported in the literature.

Keywords: nanocrystalline materials, grain size, hardness, strength, modeling, Hall–Petch relationship, grain boundary affected zone

1. Introduction
Owing to their ultrafine grains and high density of grain boundaries, nanocrystalline materials exhibit a variety of properties that are often considerably improved in comparison with the conventional microcrystalline materials, including increased strength (or hardness), superior wear resistance, improved low temperature superplastic formability, etc. The grain size of materials has a significant effect on the mechanical properties. Microcrystalline materials obey the well-known Hall–Petch relationship which can be expressed as,

$$\sigma_{\text{hp}} = \sigma_o + kd^{-1/2}, \quad (1)$$

where $\sigma_{\text{hp}}$ is the yield strength, $\sigma_o$ is the friction stress below which dislocations cannot move in a single crystal, $k$ is a constant called ‘locking parameter’ representing the relative hardening contribution of the grain boundaries, and $d$ is the average grain diameter.

Experimental data available in the literature indicate that the strength or hardness of nanocrystalline materials cannot be simply predicted by extrapolating the Hall–Petch relationship to the nanoscale grain size of about 10 nm [1]. It has been shown that the Hall–Petch slope in the nanocrystalline domain is lower than that in the microcrystalline (conventional) range of grain sizes, and the slope decreases significantly for grain sizes below a critical value. In fact, some researchers have reported a negative slope, or a decrease in flow stress with decreasing grain size, i.e. ‘grain size softening’ (also called the inverse Hall–Petch relationships) [2]. A number of theories and models have been developed to understand the deviation of the strength from the empirical Hall–Petch equation [2–19]. Recent molecular dynamics (MD) simulations have improved our understanding of the deformation of nanocrystalline materials [13]. The MD simulations in nanocrystalline copper [20–23], aluminum [11, 24], nickel [22, 23] and cobalt [25], where an inverse Hall–Petch effect was observed [11, 17, 20, 21, 24–26], revealed the difference between the grain interior and the grain boundary regions where most of the deformation occurred due to the inter-grain deformation (sliding) mechanism [20, 22, 23, 27, 28]. It was further observed that the volume fraction of the total grain boundaries, which increases with decreasing grain size [29], increases under straining conditions, indicating an expansion of the grain boundary regions during straining [11]. The nanocrystalline materials have been considered as a three-component composite
consisting of grains, grain boundaries and triple junctions, where three-grain boundaries meet [30], or a four-component composite consisting of crystallite (grain interior), grain boundaries, triple lines and quadruple nodes [31]. For the sake of simplicity, the nanocrystalline materials could be approximated as a composite with two phases only—
grain interior and grain boundary [32–35], since all plastic deformation was observed to be accommodated in the grain boundary and no intra-grain deformation occurred [22, 23]. The grain phase then represents the grain cores while the matrix phase represents a blend of grain boundaries, triple junctions and/or quadruple nodes. The results of MD simulations provide a basis for identifying the role of the grain interior and grain boundary during plastic deformation, and a potential for predicting the strength of nanocrystalline materials via composite models or the rule of mixtures [30, 31, 33]. It was also shown that the results of the finite element calculations are almost the same as those obtained using the rule of mixtures [31].

Based on the above consideration a coherent polycrystal model was proposed in which the nanocrystalline materials were regarded as a coherent precipitate strengthened two-phase alloy [6]. Recently, based on the high-resolution TEM observation of grain boundaries in an Al–Mg alloy produced by a severe plastic deformation process where the grain boundary interface showed curvature and undulations, Yang and Ghosh [36] developed a model of ‘grain boundary mantle shear zone power law creep’ to calculate the strength at various strain rates. Similarly, Dao et al. [37] observed that nano-twinned Cu after tensile testing showed dislocation pile-ups along twin boundaries (TBs) and curved TBs; a region within a few nanometers of a TB was often heavily influenced by the dislocations along the TBs. Each of the TBs may be treated as a special grain boundary, where the plastic deformation in copper with nanoscale twins is concentrated in the vicinity of the TBs. High resolution TEM imaging directly confirmed the presence of partial dislocation dissociation, dislocation pile-up and TB bending. Based upon these observations they proposed a model of ‘twin boundary affected zone (TBAZ)’, where the region adjoining the twin boundaries, considered to be elastically strained, is different from the crystal interior. The TBAZ is plastically softer than the predominantly elastic crystal interior region between TBAZs. The grain boundaries (or twin boundaries) in the above models were basically considered as a continuous matrix and the grains were embedded in the grain boundary matrix. These recent observations and models basically support the two-phase coherent polycrystal model [6]. Therefore, the coherent polycrystal model [6] involving the dependence of strength on grain sizes in the nanoscale range will be considered in this study.

Experimental observations have also revealed that intragranular dislocation sources like the Frank–Read source cease to operate in nanocrystalline materials and result in high strength and reduced plasticity in these materials. The traditional dislocation theories may no longer be applicable to the deformation behavior of nanocrystalline materials. Consistent with the recent experimental observations or models mentioned above, atomistic simulations also suggested that dislocations nucleated at grain boundaries (GBs) carry out plastic deformation in the nanocrystalline regime; once nucleated, these dislocations travel across the grains and are eventually absorbed in the opposite grain boundary [38–40]. In a very recent investigation on the plastic deformation recovery in nanocrystalline aluminum and gold films, it was reported that the enhancement of recovery rate could be due to the reduction in pinning sites caused by redistributions of grain boundary impurities during annealing [41]. It was further stated that ‘the strain recovery results from the combined effect of a small mean grain size and inhomogeneities in the microstructure (variations in the size and orientation of individual grains and structure of GBs); the small grain size precludes intragranular dislocation sources, whereas variations in the microstructure lead to plastic deformation (through dislocations originating from GBs) in relatively larger grains and elastic accommodation in smaller grains’.

‘Hence, in exploring plastic deformation of nanocrystalline materials, it might be necessary to consider not only the average size of the microstructure but also the inherent variations’. This suggests that not only the average grain size but also the inherent variation (or distribution) of the grain size should be taken into consideration [41].

This investigation was, therefore, aimed at describing the dependence of strength (or hardness) of nanocrystalline materials on the grain size, based on the Hall–Petch relationship and coherent polycrystal model via further integrating a log-normal distribution of grain sizes.

2. Modeling

In the coherent polycrystal model [6], the grain phase was assumed to be a perfect crystallite while the grain boundary phase contained potential defects (e.g. dislocations, vacancies, impurity atoms, etc) and was relatively narrow. The strengthening effect of the grain boundary matrix by the coherent precipitates, i.e. by the grain phase, may be expressed as,

$$\sigma_{\text{co}} = \sigma_{\text{gb}}^0 + k_{\text{co}}[(d - \Delta)/d]^3,$$

(2)

where $\sigma_{\text{co}}$ and $\sigma_{\text{gb}}^0$ are the strength of the nanocrystalline material and the grain boundary, respectively, $k_{\text{co}}$ is a material parameter representing the measure of the strengthening effect of grain cores on the grain boundary matrix, and $\Delta$ is the grain boundary affected zone (or mantle shear zone) or effective grain boundary thickness [6, 25, 36, 37]. Since there is a large volume of experimental data available as hardness values, it is convenient to convert the strength values to hardness values using the method given in [42].

$$H_0 = 3\sigma_0,$$

(3)

$$K = 3k.$$

(4)

Then the equivalent equation for the grain size dependence of the hardness in the coherent polycrystal model may be expressed as,

$$H = H_{\text{gb}}^0 + K_{\text{co}}[(d - \Delta)/d]^3,$$

(5)
where $H$, $H_{gb}^c$ and $K_{co}$ are the corresponding terms to those specified in equation (2). That is, $H$ and $H_{gb}^c$ are the hardness of the nanocrystalline material and the grain boundary, respectively, and $K_{co}$ is a material parameter that reflects the measure of the hardening effect of grain cores on the grain boundary matrix. There will then be a critical grain size, $d^*$, above which the deformation mechanism changes from the coherent polycrystal mechanism to the Hall–Petch type strengthening mechanism according to equation (1).

Song et al [6] also reported that $\Delta$ is associated with the average atomic diameter ($D_m$) of the major elements in the nanocrystalline materials, given by,

$$\Delta = 84D_m - 21.7,$$  
(6)

where both $\Delta$ and $D_m$ are in nm. It follows that the larger the atomic diameter, the larger the effective grain boundary thickness.

As suggested by Rajagopalan et al [41], the inherent variations in grain sizes in nanocrystalline materials should be taken into account in exploring their plastic deformation. We consider the grain size distribution which follows a log-normal function. To facilitate the analysis, the following assumptions similar to those specified in [4, 8] are used.

1. Polycrystals with a relatively large average grain size ($d > d^*$) follow the classical Hall–Petch relationship, equation (1).
2. The strength $\sigma_{co}$ or hardness $H$ of the material with very small grains complies with the coherent polycrystal model, i.e. equation (2) or (5).
3. The volume of grains obeys a certain distribution, $f(v)$, then the mean volume of all the grains, $m_v$, is given by,

$$m_v = \int_0^\infty v f(v) \, dv,$$  
(7)

or expressed in terms of the grain diameter, $D$, the mean grain size/diameter of all the grains, $d$, becomes,

$$d = \int_0^\infty D g(D) \, dD,$$  
(8)

where $g(D)$ is the grain diameter distribution. The mean volume of the grains may be further expressed as,

$$m_v = \kappa d^3,$$  
(9)

where $\kappa$ is a geometrical shape factor considered to be 1 in this analysis.

When the grain size decreases to the critical grain size, it is assumed that the conventional Hall–Petch mechanism associated with dislocation glide and pile-ups switches to the coherent polycrystal mechanism. At $d = d^*$,

$$\sigma_{hp} = \sigma_{co},$$  
(10)

where $\sigma_{hp}$ and $\sigma_{co}$ are described by equations (1) and (2), respectively.

For mean grain sizes different from $d^*$, the yield stress, after averaging via combining the Hall–Petch response with the coherent polycrystal mechanism, may be expressed as,

$$\langle \sigma_{\text{un}} \rangle = F_{hp} + F_{co},$$  
(11)

where

$$F_{hp} = \frac{1}{m_v} \int_{v^*}^\infty \sigma_{hp} v f(v) \, dv,$$  
(12)

and

$$F_{co} = \frac{1}{m_v} \int_0^{v^*} \sigma_{co} v f(v) \, dv,$$  
(13)

where $F_{hp}$ stands for the contribution of the Hall–Petch mechanism and $F_{co}$ represents the contribution of the coherent polycrystal mechanism to the overall strength. The critical volume corresponding to the transition in the mechanisms may be estimated from,

$$v^* = \kappa (d^*)^3.$$  
(14)

Equation (11) is a unified equation representing the relationship between the yield stress and the grain size with a certain grain size distribution. The yield stress of materials with a mean grain size from the nanoscale to micro-scale regime can thus be calculated from equation (11). Likewise, an equivalent equation for the grain size dependence of hardness may be expressed as,

$$\langle H_{\text{un}} \rangle = F_{hp} + F_{co},$$  
(15)

$$F_{hp} = \frac{1}{m_v} \int_{v^*}^\infty H_{hp} v f(v) \, dv,$$  
(16)

and

$$F_{co} = \frac{1}{m_v} \int_0^{v^*} H_{co} v f(v) \, dv,$$  
(17)

where $\langle H_{\text{un}} \rangle$, $F_{hp}$ and $F_{co}$ are the corresponding terms specified in equation (11).

4. The volume of grains follows a log-normal distribution, which has been observed to be reasonable in many metals, alloys and ceramic systems [4, 5, 43]. Hence, the log-normal distribution is chosen to represent the grain size distribution in the present investigation and may be expressed as,

$$f(v) = \frac{1}{v \sqrt{2\pi} s_{lnv}^2} \exp \left\{ -\frac{(\ln v - m_{lnv})^2}{2s_{lnv}^2} \right\},$$  
(18)

where $m_{lnv}$ and $s_{lnv}$ are the mean value and standard deviation of ln $v$, respectively. $m_v$ is the mean volume of all the grains, and according to equation (7) it is given by,

$$m_v = \int_0^\infty v f(v) \, dv = \exp[m_{lnv} + (s_{lnv})^2/2].$$  
(19)
Hardness, GPa

| Hardness (GPa) | Experimental | Calculated |
|----------------|--------------|------------|
| 0.1            | 0.5          | 0.3        |
| 0.2            | 0.7          | 0.5        |
| 0.3            | 0.9          | 0.7        |
| 0.4            | 1.1          | 0.9        |

Figure 1. The hardness calculated from equation (15) as a function of grain sizes in the form of $d^{-1/2}$ for the nanocrystalline Zn, in comparison with the experimental data [1].

3. Applications of the model and discussion

3.1. Applications

The developed model can be applied to a number of metallic systems for which the experimental data are available in both nanoscale and micro-scale regimes. Like the Hall–Petch equation, the parameters in the coherent polycrystal model can be determined from the experimental data according to equation (2) or (5).

Koch and Narayan [1] and Narayan et al [44, 45] reported that the hardness of the nanocrystalline metal Zn deviates from the Hall–Petch relationship when the grain size is less than about 11 nm and exhibits an inverse Hall–Petch effect. The Hall–Petch parameters from their experimental data are: $H_o = -0.51$ GPa and $K = 5.32$ GPa $\cdot nm^{1/2}$ [1]. As suggested in [6], $\Delta$ may be estimated from equation (6), which leads to $\Delta = 4.1$ nm using the $D_\infty$ value of Zn given in [46]. The two parameters in the coherent polycrystal model, $H^*_pb$ and $K_{co}$, may be obtained from the experimental data in the nanoscale regime in terms of equation (5): $H^*_pb = 0.52$ GPa, and $K_{co} = 2.02$ GPa. Using the above five parameters, the hardness from the micro-scale to the nanoscale regime can be estimated from equation (15). Matlab software was used to calculate the numerical values from equations (15) to (17). The critical grain size is obtained as $d^* = 11.4$ nm using equation (10). The corresponding maximum hardness is $H^* = \langle H_{tot}(d^*) \rangle = 1.07$ GPa, which is very close to the experimental value $H \approx 1.04$ GPa. Figure 1 shows the calculated hardness values as a function of the grain size for the nanocrystalline Zn. It is seen that, after the grain size distribution is taken into consideration, a gradual variation of the hardness values is observed. Furthermore, the hardness values calculated from equation (15) are in agreement with the experimental data.

Figures 2(a) and (b) show the calculated hardness curves as a function of $d^{-1/2}$, in comparison with the experimental data for the Ni–P [47, 48] and TiAl [49] systems, respectively. In these alloy systems, the parameters of the Hall–Petch relationships were given in [47, 49], while the parameters of the coherent polycrystal model were presented in [6]. Again, the calculated hardness curves show good agreement with the experimental data, and the curves exhibit a gradual transition from the Hall–Petch mechanism to the coherent polycrystal mechanism. The present analysis, via the integration of the Hall–Petch mechanism with the coherent polycrystal mechanism and the consideration of a lognormal grain size distribution, can thus be used to describe the grain size dependence of hardness from micro-sized grains to nanosized grains.

The gradual transition process may be understood in the following way. In a sample with an average grain size close to the critical grain size, $d^*$, some grains would have sizes larger than $d^*$, and others smaller than $d^*$. It is anticipated that there would co-exist two deformation mechanisms at the same time [41]. The grains with larger sizes obey the traditional Hall–Petch relationship related to the dislocation glide and pile-up strengthening mechanism, whereas the rest of the grains with smaller sizes behave according to the coherent polycrystal mechanism. The overall macro-effect of the deformation of the sample would be the superposition of the two mechanisms. With decreasing average grain size, the fraction of the grains deformed via the dislocation glide and pile-up strengthening mechanism decreases, while the fraction of the grains deformed according to the coherent polycrystal mechanism increases. This gives rise to an overall variation from strengthening to softening in the polycrystalline materials.
3.2. Effect of effective grain boundary thickness

In equation (11) or (15), there are five material parameters, \( \sigma_0, k, \sigma_0', k_{gb} \) (or in the case of hardness, \( H_0, K, H_0', K_{gb} \)), and the effective grain boundary thickness, \( \Delta \). It should be noted that the concept of effective grain boundary thickness, or grain boundary affected zone, or mantle shear zone, or twin boundary affected zone, has also been considered recently by a number of researchers [21, 25, 36, 37]. Since \( \Delta \) was suggested to be proportional to the atomic diameter [6], or the twin boundary affected zone was suggested to span 7–10 lattice parameters in thickness (i.e. 2.5–3.6 nm in the nanocrystalline copper) [37], the deformation characteristics of nanomaterials would be influenced by the value of \( \Delta \).

Figure 3(a) shows the calculated values of hardness, \( \langle H_{gb} \rangle \), of nanocrystalline Zn according to equation (15) for different values of \( \Delta \) while keeping other parameters unchanged. Similar results are given in figures 3(b) and (c) for two other nanocrystalline materials, Ni–P and TiAl, respectively.

Figure 3. The calculated values of hardness from equation (15) with different \( \Delta \) values. (a) Zn, (b) Ni–P and (c) TiAl.

As a result, when the effective grain boundary thickness \( \Delta \) (or the grain boundary affected zone) decreases, the deformation becomes more limited and difficult, thus the hardness in the nanoscale regime increases with decreasing \( \Delta \). With an appropriate selection of \( \Delta \) value based on the atomic diameter or lattice parameter of the principal element in the nanocrystalline material [6, 37], the proposed model can be used to adequately describe the dependence of hardness over a wide range of grain sizes. For example, in the case of nanocrystalline Zn, \( \Delta = 4.1 \) nm, the model gives a reasonable description of the relationship between the hardness and the grain size, as shown in figure 1.

The effect of \( \Delta \) on the critical grain size is shown in figure 4 for different nanocrystalline materials. A linear relationship is observed, which may be expressed as,

\[
d^* = d^*_0 + \alpha \Delta, \tag{20}
\]

where \( d^*_0 \) and \( \alpha \) are constants. When the grain boundary affected zone is absent (i.e. \( \Delta = 0 \)), \( d^*_0 = d^* \), the critical grain size. Figure 5 shows the maximum hardness as a function of \( \Delta^{-1/2} \). A linear variation of the maximum hardness with \( \Delta^{-1/2} \) is also observed, i.e.

\[
H^* = H^*_0 + \beta \Delta^{-1/2}, \tag{21}
\]

where \( H^*_0 \) and \( \beta \) are constants. \( \alpha \) represents the effect of the effective grain boundary thickness on the critical grain size, while \( \beta \) represents the effect of the effective grain boundary thickness on the maximum hardness. Equations (20) and (21) show that the thinner the effective grain boundary in the nanocrystalline materials, the smaller the critical grain size, and the higher the maximum hardness of the materials. This can be understood by noting that a smaller critical grain size due to a smaller effective grain boundary thickness is equivalent to extending the range valid for the Hall–Petch equation to a smaller grain size. Since the hardness or strength increases with decreasing grain size in the Hall–Petch relationship, the maximum hardness \( H^* \) at the smaller critical grain size becomes higher. The parameters based on equations (20) and (21) for the three materials are listed in table 1. It appears that \( \alpha \) is relatively insensitive to the material systems as it changes only between 0.73 and 1.09,
while $\beta$ is strongly dependent on the material, indicating that the maximum hardness $H^*$ is a strong function of the effective grain boundary thickness $\Delta$.

### 4. Conclusions

1. The effect of grain size on the strength or hardness was attributed to two deformation mechanisms associated with inhomogeneities in the microstructure: (i) a fraction of larger grains deform by a dislocation glide and pile-up process according to the Hall–Petch relationship, and (ii) the smaller grains deform by the coherent polycrystal mechanism (a precipitate-like strengthening mechanism). With decreasing average grain size, the fraction of deformation associated with the dislocation glide decreases, and the fraction of deformation via the coherent polycrystal mechanism increases, giving rise to an overall change in the response from strengthening to softening of the material.

2. By combining the conventional Hall–Petch relationship for larger grains with the coherent polycrystal mechanism for smaller grains and introducing a log-normal distribution of grain sizes, a model was developed to characterize the grain size dependence of hardness (or strength) in nanocrystalline materials.

3. The transition from the Hall–Petch relationship to the coherent polycrystal mechanism was shown to be a

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**Table 1.** Parameters in equations (20) and (21) for different nanocrystalline materials.

| Materials | $d^* = d^*_o + \alpha \Delta$ | $H^* = H^*_o + \beta \Delta^{-1/2}$ |
|-----------|-------------------------------|----------------------------------|
| Zn        | $d^*_o$ (nm) 8.35 $\alpha$ 0.73 $H^*_o$ (GPa) 1.88 $\beta$ (GPa · nm$^{-1/2}$) 9.83 |
| Ni–P      | $d^*_o$ (nm) 5.23 $\alpha$ 1.09 $H^*_o$ (GPa) 6.01 $\beta$ (GPa · nm$^{-1/2}$) 1.20 |
| TiAl      | $d^*_o$ (nm) 15.25 $\alpha$ 1.01 $H^*_o$ (GPa) 4.63 $\beta$ (GPa · nm$^{-1/2}$) 17.46 |

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**Figure 4.** The critical grain size, $d^*$, as a function of the effective grain boundary thickness, $\Delta$. (a) Zn, (b) Ni–P and (c) TiAl.

**Figure 5.** The maximum hardness corresponding to the critical grain size versus the effective grain boundary thickness in the form of $\Delta^{-1/2}$. (a) Zn, (b) Ni–P and (c) TiAl.
gradual process, and not an abrupt change from one mechanism to the other.

4. The effective grain boundary thickness representing the grain boundary affected zone was observed to have a significant effect on the critical grain size and the hardness (or strength) in the nanoscale regime.

5. The critical grain size in the nanocrystalline materials was observed to increase linearly with increasing effective grain boundary thickness.

6. The maximum hardness in the nanocrystalline materials was found to increase with decreasing effective grain boundary thickness (\(\Delta\)) in the form of \(\Delta^{-1/2}\).

7. The calculated hardness values were observed to be in good agreement with the experimental data on the grain size dependence of the hardness from the micro-scale to nanoscale regime.

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