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Hardness length-scale factor to model nano- and micro-indentation size effects

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ABSTRACT

In this paper, we show that nano- and micro-indentation hardness data can be represented adequately by the strain gradient plasticity (SGP) theory if the uniformity of the dislocation spacing is taken into account. To give relevant information on the plastic deformation process, we suggest to use a hardness length-scale (HLS) factor equal to $\text{Ho} \cdot \sqrt{h*}$, where Ho is the macro-hardness and h^* the characteristic scale-length deduced from the hardness–depth relation of the SGP theory. Theoretically, the HLS factor is proportional to both the shear modulus and the Burgers vector, depending on the dislocation spacing. Applied to various crystalline metals, the representation of the experimental HLS factor as a function of the theoretical one shows two distinct linear behaviours related to the micrometer and nanometer depth regimes associated with a uniform dislocation organisation beneath the indenter and with dislocations located at the vicinity of the indenter tip in a largest plastic zone, respectively.

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1. Introduction

In indentation, it is well recognized that the hardness value of materials could be independent of load, it could increase or decrease with load, and it could show a complex variation with load changes [1]. At low loads the effect of the surface layers with different material properties is more pronounced. However, as the indentation depth increases, the bulk effect becomes more dominant and eventually there is no change in the hardness value with the load. In general, this load-dependence of micro-hardness is known as the indentation size effect (ISE). This phenomenon has been associated with various causes such as work hardening, roughness, piling-up, sinking-in, shape of indenter, surface energy, varying composition and crystal anisotropy, which have been all discussed extensively by Cheng and Cheng [2]. Many relationships, dating from 1885 to the present, have been suggested to describe the load-dependence of hardness. In recent years, Ma and Clarke [3] and Nix and Gao [4] have introduced the concept of strain gradient plasticity (SGP) based on Taylor's dislocation theory. Besides, Nix and Gao [4] have shown that the ISE behaviour of crystalline materials can be accurately modelled using the concept of geometrically necessary dislocations (GND). The authors based their reasoning on the experimental law needed to advance a mechanism-based theory of strain gradient plasticity. For a glassy polymer, Lam and

Chong [5] describe the hardness–depth variation using a SGP theory, which differs from that of Nix and Gao [4] in the manner in which dislocations of the global plastic deformation process are separated. Abu Al-Rub and Voyiadjis [6] have developed a similar relationship to determine a material length-scale using microhardness results. The relation between micro-indentation hardness and indentation depth variables, macro-hardness and length-scale parameters involves an exponent, which can vary between 1 and 2 corresponding to the relations of Lam and Chong [5] and Nix and Gao [4], respectively.

On the other hand, Nix and Gao's model of GND underneath a sharp indenter is well recognized and nowadays largely used to represent micro-indentation hardness data. The relation established by Nix and Gao [4] between micro-indentation hardness, *H*, and indentation depth, *h*, is given by:

$$\left(\frac{H}{\text{Ho}}\right)^2 = 1 + \left(\frac{h^*}{h}\right) \tag{1}$$

where Ho is generally called the macro-hardness and corresponds to the hardness that would arise from the statistically stored dislocations alone, in the absence of any geometrically necessary dislocations. The distance, h^* , is the characteristic scale-length which characterizes the depth-dependence of hardness.

Huang et al. [7], after analysing numerous works [8–16] have reported that nano-indentation hardness data do not follow Eq. (1). For the majority of these authors, nano-indentation and microindentation typically refer to indentation depths below and above 100 nm, respectively. On the other hand, in order to explain the dis-

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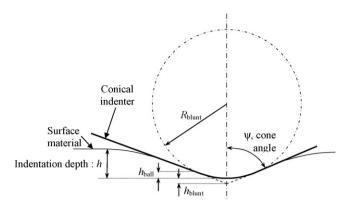


Fig. 1. Scheme of a conical indenter showing the tip blunt ($R_{\rm blunt}$) and the characteristic parameters for describing tip radius effects: $h_{\rm blunt}$ to compare a perfect cone to an indenter and $h_{\rm ball}$ corresponding to a ball indentation depth.

crepancy between Eq. (1) and nano-indentation hardness data, the authors advanced two factors linked to the effect of the indenter tip radius and the storage volume for the GND. As shown in Fig. 1, the effect of the tip radius greatly depends on the blunt tip indenter. For a new indenter, the tip radius ($R_{\rm blunt}$ in Fig. 1) is generally close to 50 nm but it can reach more than 500 nm when the indenter is considered as defective. Geometrical considerations allow the calculation of $h_{\rm blunt}$ and $h_{\rm ball}$, equivalent to a ball penetration, as a function of the tip radius, $R_{\rm blunt}$, and the cone semi-angle, ψ , measured at the tip indenter. The calculations indicate that:

$$\begin{cases} h_{\text{blunt}} = R_{\text{blunt}} \cdot \left(\frac{1}{\sin \psi} - 1\right) \neq 0.06R_{\text{blunt}} \\ h_{\text{ball}} = R_{\text{blunt}} \cdot (1 - \sin \psi) \neq 0.06R_{\text{blunt}} \end{cases}$$
 (2)

where ψ is equal to 70.3°, which gives a cone area function equivalent to those obtained with a Berkovich and a Vickers pyramid indenters.

For a new indenter, h_{blunt} and h_{ball} are both equal to 3 nm which can be often neglected in comparison with the experimental indentation depths. h. For a blunted indenter, it is not so obvious since the two corrected depths are equals to 30 nm which are relatively high values compared to typical nano-indentation depth values. However, the indenter tip radius is usually taken into account through a corrected indented area for the calculation of the actual nanoindentation depth into the material. According to Huang et al. [7], the indenter tip radius effect cannot alone explain the nanoindentation size effects. As a consequence, the difference should be mainly connected to a factor representative of the dislocations network and jointly of the storage volume of the dislocations. That is why, in order to represent nano-indentation hardness data, Huang et al. [7] suggest the consideration of a maximum allowable GND density allowing the representation of the hardness data below and above the limit value between nano- and micro-hardness data.

Fig. 2 represents the model of Huang et al. [7] applied to hardness data resulting from indentation experiments performed by Feng and Nix [14] on magnesium oxide (MgO). On this figure, the authors have plotted Nix and Gao's model which describes adequately the hardness data for indentation depths higher than approximately 200 nm. We can notice that this limiting indentation depth value is twice the typical value of 100 nm admitted by some authors for delimiting nanometer and micrometer depth regimes. As a first observation, the limiting indentation depth does not seem to have a fixed value and depend on the material properties and the indenter tip radius. In addition, the model of Huang et al. [7] is not able to represent adequately the hardness—depth variation in the depth-range located between the two indentation scales of measurement, i.e. for indentation depths between 100 and 300 nm for the analysed mate-

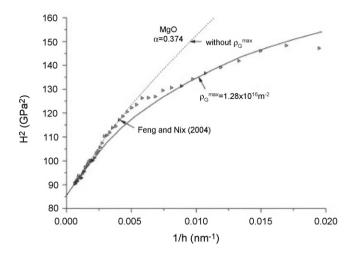


Fig. 2. Square of indentation hardness, H^2 , versus the reciprocal of indentation depth, 1/h, given by the finite element analysis for MgO. Nix and Gao's model is also shown (without accounting for GND_{max}) [7].

rial, as shown in Fig. 2. That is why, to overcome such a discrepancy at the transition stage between nanometric and micrometric scales, Zong et al. [17] suggested that the hardness–depth data could be modelled in two separate zones by means of two straight lines represented by Eq. (1). Subsequently the authors give two sets of characteristic indentation parameters (Ho and h^*) respectively to describe nano- and micro-indentation experiments. However Zong et al. [17] do not give additional physical explanations to interpret jointly the macro-hardness values (Ho_{micro} and Ho_{nano}) and the characteristic scale-lengths ($h^*_{\rm micro}$ and $h^*_{\rm nano}$).

In the present paper, we have reanalysed various nano- and micro-indentation hardness data obtained on crystalline metals. We show that the hardness-depth relationship, originally established by Nix and Gao [4], is in reality well adapted to represent the bi-linear indentation behaviour on the two scales of measurement if some precautions are taken in accordance with the observations of Durst et al. [15] concerning the size of the plastic zone. In order to render more pertinent the comparison of the two couples (Ho and h*) deduced from nano- and micro-indentation experiments, we suggest a new indentation parameter called hardness length-scale (HLS) factor since it is capable to describe the uniformity of the dislocation organisation beneath the indenter in the two indentation depth regimes. From an experimental point of view, this parameter is directly linked to the slope of the straight line representing the square of the hardness versus the reciprocal indentation depth. In practice, HLS factor is equal to $Ho\sqrt{h*}$. Its experimental value is then compared to the theoretical product $\mu\sqrt{b}$ resulting from the SGP theory, where μ is the shear modulus and b the Burgers vector. The ratio between these two products is a constant which is only linked to the dislocation spacing and jointly to the size of the plastic zone. In micro-indentation, this factor corresponds to that of Nix and Gao's model confirming, therefore, the SGP theory. In nano-indentation, the SGP factor should take into account the non-uniformity of the dislocation spacing underneath the indenter, or the largest plastic zone, to be valid. As a main result, we clearly demonstrate that the difference between nano- and microindentation comes from the dislocation spacing under the indent. Then, to jointly interpret nano- and micro-indentation hardness data, we suggest the calculation of the hardness length-scale factor which gives more information on the plastic deformation process itself. Note that this original approach involves the maximum allowable GND density in the same way as the model of Huang et al. [7] and the model of Durst et al. [15].

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