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Comment on "Energetically evaluated load-indentation measurements of different classes of material" by B. Rother

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It is shown that the energetically evaluated load-indentation measurement procedure introduced by Rother and Dietrich is equivalent to the analysis of the force-indentation depth-curve by means of the well known Bernhardt formula. A disadvantage of the proposed "differential load feed" (DLF) procedure is that differentation enlarges the scatter of the experimental curves. Deviations of the indenter tip from its ideal shape and surface roughness also influence the experimental results in the case of DLF analysis in a manner as one observes for these methods usually used to analyse force-indentation depth-curves.

1. Introduction

Hardness H is a widely accepted measure to characterize the mechanical properties of solid surfaces or near surface regions. It is defined as the resistance to deformation due to indentation [1] and will be calculated by

$$H = \frac{F}{A} \quad (1)$$

where F is the indentation force and A is a suitably chosen part of the indenter area. The registration of a force-indentation depth-curve gives the most objective, most reliable and most sensitive method to estimate the hardness [2, 3], for example to determine the so-called universal hardness HU [4, 5]. For the often used Vickers hardness the connection between indenter area A of Equation 1 and indentation depth s is given by

$$A = ks^2 \quad (2)$$

where $k = 26.43$ is the geometry factor for a Vickers pyramid.

Initially it was hoped that hardness would be a material parameter independent of the experimental conditions, and this is true for relatively high indentation depths. However, the improvement of the sensitivy of measuring devices has shown that hardness defined by Equation 1 is indentation depth and force dependent for low indentation depths [6–8]. (This is also true for low forces in the case of conventional hardness measurements, where the indentation diagonal after unloading is used to estimate the indentation area A [9, 10].)

Therefore many authors have tried to describe this indentation dependence of the hardness by suitable analytical equations. The parameters of such an equation will be considered as the "true material parameters", characterizing the mechanical behaviour. Examples are Meyer's law (e.g. [9]), Bernhardt's formula [11, 12], Thomas' equation [13], the archtangen function [14], and, recently, an energetic measure [15]. The last one, and its connection with the usually used hardness, is discussed in the following.

2. Energetic hardness measure for an ideal indenter

Rother and Dietrich [15] proposed that the indentation procedure can be described energetically by a sum of two terms

$$W = e_V s^3 + e_A s^2 \quad (3)$$

where e_V is a volume related part and e_A represents an area related part with the assumption that "a linear relation (exists) between the indentation depth and the dimension of the energy densification zone under the penetrating indentor" [16]. The indentation force F follows by the first derivative of Equation 3

$$F = 2e_A s + 3e_V s^2 \quad (4a)$$

This equation agrees exactly with the force-deformation-equation already given by Bernhardt [11] for conventional hardness tests and first applied by Fröhlich *et al.* [6] for the analysis of recording hardness measurements

$$F = a_1 s + a_2 s^2 \quad (4b)$$

Also, Fröhlich *et al.* [6] tried to interpret Equation 4b energetically. Integration gives Equation 3 with

$$a_1 = 2e_A; \qquad a_2 = 3e_V \quad (4c)$$

The first term of Equation 3 was identified as a volume part of deformation. This is the same interpretation as was later given by Rother [16]. The second term was discussed as the energy part to increase the surface of the indentation. However, a comparison with known surface energies of solids does not give a satisfactory agreement. Therefore Fröhlich *et al.* [6] have used Equation 4b as a formal description of the force-indentation depth-dependence of indentation measurements. Rother [16] interprets this second term of Equation 3 as the energy to produce a densification zone. This also appears to be a formal description, as until now there has been no possibility to examine it by an independent measurement of this magnitude. Additionally, it should be borne in mind that densification would be expected only for amorphous materials and could be proved for fused silica by the increase of the diffraction index in the neighbourhood of the indentation [17, 18].

Rother [16] proposes to analyse the force-indentation depth-curves by a plot of dF/ds versus s (differential load feed, DLF), since

$$\frac{dF}{ds} = \frac{d^2W}{ds^2} = 2e_A + 6e_V s \tag{5a}$$

must give a straight line. However, the aim of a linear relation can also be reached by a plot of F/s versus s, as Equation 4b yields

$$\frac{F}{s} = a_1 + a_2 s \tag{5b}$$

in agreement with Equation 5a, proposed already in [6]. The first proposal has the disadvantage that the differentation procedure increases the scatter of the curves, so that "a specially developed averaging and smoothing procedure was applied" [16], for details see [19]. Moreover, the discontinuities in the load-penetration depth curves, characteristic of, for example, depth sensitive hardness measurements of polycrystalline metals ("effects of second order" in [14]) are increased significantly by the DLF-procedure. The use of Equation 5b avoids this difficulty.

An experimental confirmation of one of the equivalent Equations 3, 4 or 5 includes simultaneously the statement that hardness increases with decreasing indentation depth, as Equations 1, 2 and 4b give

$$H = \frac{1}{k}\left(\frac{a_1}{s} + a_2\right) \tag{6}$$

This formula was used, amongst others, by Thomas [13] to describe the so-called indentation size effect (ISE) without a direct connection to the force-indentation depth-relation Equations 4a or 4b. A procedure to determine the constants a_1 and a_2 directly from measured hardness values is a H versus $1/s$ plot, which must give a straight line if Equations 3 or 4 describe the material behaviour correctly.

In summary, one can say that a straight line in DLF and an ISE in a hardness versus indentation depth plot are expressions of the same material behaviour. Both methods correctly applied yield the same material parameters. Therefore it is not correct to state that "linear DLF ranges are considered as proof of a constant resistance of the probed material against the penetration" [16], whereas contrary to this response the HU values show a typical decrease with increasing penetration depth. These statements are not contradictory, as they follow from the same deformation law.

It is also a result of the foregoing analysis that the part e_V of the energy sum is a measure of the indentation depth independent part a_2 of the hardness, see Equation 4c.

3. Influence of deviations from the ideal behaviour

Ideal conditions were assumed in section 2, i.e. a perfect indenter shape and an ideal smooth solid surface. In the following the influence of a deviation from these conditions is investigated.

An indentation tip imperfection increases the ideal indenter area A_{id} to its real value A_{re}

$$A_{re} = \eta_T A_{id}; \qquad \eta_T \geqslant 1 \tag{7}$$

For a rounded indenter with a tip radius R_{tip} [20, 22]

$$\eta_T = 1 + 0.158\frac{R_{tip}}{s} + 0.000574\frac{R_{tip}^2}{s^2} \tag{7a}$$

This means that the higher the tip rounding the higher is the tip imperfection factor η_T. The surface roughness of the solid changes the effective indenter area from A_{re} to its value A_{ef} in such a way that the effective area can be lower or higher [21]:

$$A_{ef} = \frac{1}{\eta_R}A_{re}; \qquad \eta_R \underset{>}{<} 1 \tag{8}$$

Therefore Equation 1 yields the true hardness value

$$H = \frac{F}{A_{ef}} = \frac{\eta_R}{\eta_T}\frac{F}{A_{id}} = \frac{\eta_R}{\eta_T}H_{fic} \tag{9a}$$

with a fictive hardness H_{fic} calculated on the basis of ideal conditions [20]:

$$H_{fic} = \frac{F}{A_{id}} = \frac{F}{26.43\,h^2} \tag{9b}$$

H_{fic} is identical to the universal hardness HU for Vickers indentation. Naturally both the tip factor η_T and the roughness factor η_R can depend on the indentation depth s, see, for example, Equation 7a.

The material behaviour is given by the true hardness $H(s)$ of Equation 9a. So the measured $F(s)$ curve follows from

$$F(s) = \frac{\eta_T(s)}{\eta_R(s)}A_{id}(s)\,H(s) = k\frac{\eta_T(s)}{\eta_R(s)}s^2\,H(s) \tag{10}$$

where the geometry relation Equation 2 for the ideal indenter area was used. The Rother analysis [16] requires

$$\frac{dF}{ds} = k\frac{d}{ds}\left(\frac{\eta_T}{\eta_R}\right)s^2\,H(s) + 2k\frac{\eta_T}{\eta_R}s\,H(s) + k\frac{\eta_T}{\eta_R}s^2\frac{dH}{ds} \tag{11}$$

which means that the DLF also includes the tip and the roughness factors. Therefore one must expect that the parameters derived from the dF/ds versus s plot are also influenced by the deviation from the ideal behaviour. This will be shown for the material law Equation 6, which is the basis of the energetically evaluated load-indentation measurement proposed by Rother [16].

Equation 10, in combination with Equation 6, gives, by differentiation

$$\frac{dF}{ds} = \frac{d}{ds}\left(\frac{\eta_T}{\eta_R}[a_1 s + a_2 s^2]\right)$$

$$= [a_1 s + a_2 s^2]\frac{d}{ds}\left(\frac{\eta_T}{\eta_R}\right) + [a_1 + 2a_2 s]\frac{\eta_T}{\eta_R} \quad (12)$$

This is identical to Equation 5a in the ideal case $\eta_T = \eta_R = 1$, but, in general, the parameters determined by the DLF analysis are changed due to the imperfect experimental conditions. This appears to be the case also for the usually applied analysis of the hardness-indentation depth-curve. The fictive hardness is derived from the $F(s)$ curve and because of its dependence on the true hardness Equation 9a one finds

$$H_{fic} = \frac{1}{k}\frac{\eta_T}{\eta_R}\left(\frac{a_1}{s} + a_2\right) \quad (13)$$

These parameters are also determined by the analysis of H_{fic} (e.g. by a H_{fic} versus $1/s$ plot) and are influenced by the imperfection factors. In both cases it is necessary to correct the experimentally determined parameters to obtain the true material parameters.

Naturally one cannot rule out the possibility that for special cases of η_T and/or η_R one of the parameters is not influenced by the imperfection factors.

4. Conclusion

The energetically evaluated load-indentation measurement proposed by Rother and Dietrich [15] has the same basis as the analysis of the universal hardness with the Bernhardt formula. In both cases one obtains the same material parameters. They are also influenced by the experimental conditions, especially by the correction factors η_T of the tip imperfection and η_R of the roughness of the solid surface. In both cases it is necessary to correct the experimental determined parameters to obtain the true material parameters, if the factors mentioned deviate noticeably from unity.

A disadvantage of the Rother analysis is that analysable results can be expected only for ranges where the Bernhardt formula is valid. However, it is known [23] that for small indentation depths, i.e. in the so-called nano-indentation-range, deviations from this course are observed. Also, most examples given in [15, 16] show, in the DLF plot, a clear non-linear behaviour for small indentation depths. This requires a more adequate description of the indentation depth dependence of the material behaviour as the Bernhardt formula and the energy sum proposed by Rother [16], respectively, can give. A possible description yields an arctangent function as a transition function between surface and bulk hardness [14]. This proposal has the advantage that hardness is finite also for indentation depths approaching zero, whereas an exact validity of DLF also for small indentation depths would yield an infinite hardness ($H \to \infty$ for $s \to 0$ in Equation 6). Further investigations must concern the behaviour of hardness for very small indentation depths, since for recent applications the mechanical properties of the very near surface regions of a solid are important.

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