Analysis of microhardness data using the normalized power law equation and energy balance model

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It is well known that at low load the hardness is dependent on indentation load/size, which is known as the indentation size effect (ISE) [1–6]. The power law equation, generally employed to analyze the load-indentation data [1], can be expressed as:

$$P = k_1 d^{\mathrm{n}} \tag{1}$$

where, P is load, k_1 and n are materials constants and d is indentation (diagonal/diameter) size. This power law equation is also referred to in literature as Meyer's law [2–4, 6, 7]. It should be noted that Meyer's equation was originally developed for a spherical indenter, where n is directly related to the strain hardening coefficient of the material [1, 8]. Onitsch [1, 9] extended this Meyer's power law equation for nonspherical indenters and observed that in a macrohardness range n is 2.0, whereas in a microhardness range, n is less than 2, irrespective of the type of material. When n is equal to 2, the above power law equation is also quoted as Kick's law in literature [2, 10]. It has been pointed out earlier by several workers that Meyer's constant, k_1 is having a strange dimension of force/(length)$^{\mathrm{n}}$, which is dependent on the value of n [3, 11, 12]. In order to resolve this problem, Li and Bradt [3] introduced the reference indentation size corresponding to load independent hardness, whereas, Sargent [11] suggested using 10 μm indentation size corresponding to the standard hardness as a reference. Gong *et al*. [4] have proposed a modified energy balance model after realising the limitations of the existing energy and force balance model [2, 3, 12–14] for analysing the problem of the ISE and for determining true hardness i.e., the load independent hardness. The corresponding equation can be expressed as

$$P = a + bd + cd^2 \tag{2}$$

where, a is a measure of the surface residual stress and experimental error, b and c are mainly related to the surface and volume energy respectively. According to this model the true hardness, H_{T}, is found to be kc, where k is the shape factor of an indenter (e.g., k is 1.8544 and 2 for Vickers hardness and for Meyer's hardness respectively). The aim of the present paper is to analyze further the Meyer's equation and to correlate with the energy balance model. Analysis based on the proposed approach will be carried out using the experimental data available in literature.

Let us determine the hardness for an indentation using load, P and corresponding indentation area, A and indentation size, s. By inserting the expression of P from Equation 1, we can obtain the expression of hardness (H_{s}) in terms of indentation size, Meyer's constant and indenter shape factor as:

$$H_{\mathrm{s}} = \frac{P}{A} = k\frac{P}{s^2} = \frac{kk_1 s^{\mathrm{n}}}{s^2} = kk_1 s^{\mathrm{n}-2}. \tag{3a}$$

From the above equation, Meyer's constant k_1, can be written in terms of hardness as

$$k_1 = \frac{H_{\mathrm{s}}}{ks^{\mathrm{n}-2}}. \tag{3b}$$

Now inserting the expression for constant k_1 in Equation 1, we can obtain as

$$P = \frac{H_{\mathrm{s}}}{ks^{\mathrm{n}-2}} d^{\mathrm{n}} = \frac{H_{\mathrm{s}} s^2}{k}\left(\frac{d}{s}\right)^{\mathrm{n}} \tag{4}$$

or simply as, $P = K_{\mathrm{m}} D^{\mathrm{n}}$, where, $K_{\mathrm{m}} = \frac{H_{\mathrm{s}} s^2}{k} = P_{\mathrm{s}}$ as normalized Meyer's constant and $D = \frac{d}{s}$, which is defined as normalized indentation size. By this transformation, we can overcome the dimensional problem encountered in the classical Meyer's equation. Now this new Meyer's constant can be related to hardness or load for the indentation, which can be defined at any length scale. However as a natural choice, we will assume, $s = 1$ μm indentation size, so that we can recover the classical Meyer's equation in the sense of parameters but not in terms of exacts units and dimensions of Meyer's constants. The normalized Meyer's constant will have a force dimension. So the Equation 5 can be transformed to hardness equation and can be written as (using $s = 1$ μm):

$$H = H_{1\mu} D^{\mathrm{n}-2} = \frac{kK_{\mathrm{m}}}{s^2} D^{\mathrm{n}-2} = kK_{\mathrm{m}} D^{\mathrm{n}-2}. \tag{5}$$

Now we can summarise the relations obtained by normalising the classical Meyer's power law equation as:

$$P = K_{\mathrm{m}} D^{\mathrm{n}} \quad \text{or,} \quad P = P_{\mathrm{s}} D^{\mathrm{n}} \tag{6a}$$

$$\text{and} \qquad H = H_{1\mu} D^{\mathrm{n}-2}. \tag{6b}$$

Using the notation of Li and Bradt [3] for the critical indentation size as d_0^* and critical load P_{c} we can arrive from Equation 6a as:

$$P = P_c \left(\frac{d}{d_0^*} \right)^n . \quad (7)$$

While comparing this equation with the Equation 17 of Li and Bradt [3], we have noted that both the equations are identical except an extra $(2/n)$ factor, which is associated with the right hand side of their Equation 17. After analysing their approach, which is different from the present one, we found that this factor was erroneously incorporated in the normalized Meyer's equation proposed by them, though the actual numerical value may not significantly affect the analysis. However the normalized Meyer's equation, in terms of critical load and indentation size should be represented correctly by the above Equation 7.

The nature of the above Equation 6b suggests that hardness continuously decreases with the increase of load/size. Therefore, it cannot predict the transition from ISE regime to non-ISE regime. In order to determine the transition, we propose to adopt the concept of the true hardness H_T based on energy balance model. Initially, at lower load the apparent hardness will give rise to ISE, but after a critical load or indentation size, the H_A will be equal to H_T. The apparent hardness obtained from normalized Meyer's Equation 6b can be equated with the true hardness corresponding to critical indentation size, d^*. Therefore, from this above argument we can correlate the Meyer's equation with the energy balance model. The condition of equality is as follows:

$$\text{As } H_A = H_1 D^{n-2}, \quad H_T = kc;$$

therefore,

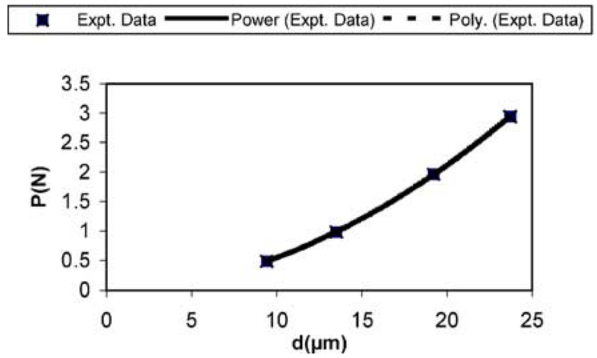
$$H_1 D^{n-2} = kc. \quad (8)$$

Now from the above condition, we can obtain the critical indentation size, d^* after which ISE should cease to exist. Putting the value of $H_1 = kK_m s^2$ and rearranging the above equation we obtain as:

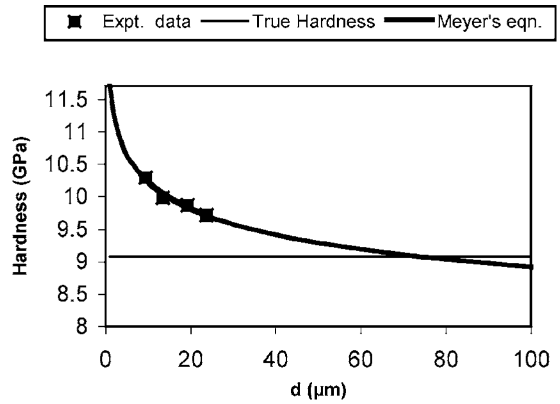
$$d^* = \left(\frac{K_m s^2}{c} \right)^{\frac{1}{2-n}} = \left(\frac{K_m}{c} \right)^{\frac{1}{2-n}} \quad \text{in } \mu\text{m as} \quad s = 1\ \mu\text{m}. \quad (9)$$

This is an important relation, which correlates between the normalized Meyer's equation and energy balance model. The implication of this equation suggests the existence of a critical length scale related to the upper bound of the ISE. Similarly, the corresponding critical load can also be determined.

Now we can analyze the indentation data obtained from AlCoCu [6] and AlCoNi [7] decagonal quasicrystals and $Mg_{32}(AlZn)_{49}$ intermetallic compound [16]. Though the Meyer's classical equation has been used to analyse in the former two cases [6, 7] but the true hardness has not been determined. Here we will be able to evaluate the true hardness and critical indentation size.Figs 1, 2, and 3show the plot of the load-indentation and hardness indentation data from Vickers microindentation experiments. It can be mentioned that the data for AlCoCu were taken upto 300 g load as the extensive cracking at higher load makes the indentation measurement inaccurate. Both the Meyer's equation and the energy balance model were fit with the experimental data satisfactorily (Figs 1a, 2a, and 3a) with regression coefficient more than 0.99. The true hardness (H_T) was obtained from the energy balance model in each case. The details of the coefficients are summarised as follows:



(a)



(b)

Figure 1 (a) Plot of the variation of load versus indentation diagonal obtained from Vickers microindentation experiment [6] of the AlCuCo decagonal quasicrystalline phase. (b) The experimental hardness data, the hardness curve from Meyer's Equation 6b, and the True hardness line obtained from energy balance model, are plotted against the indentation diagonal.

(a) For AlCoCu decagonal quasicrystalline material [6]

$$\text{Meyer's law: } P = 0.00631 d^{1.9409};$$

$$\text{Energy Model: } P = -0.00358 + 0.0096d + 0.0049d^2$$

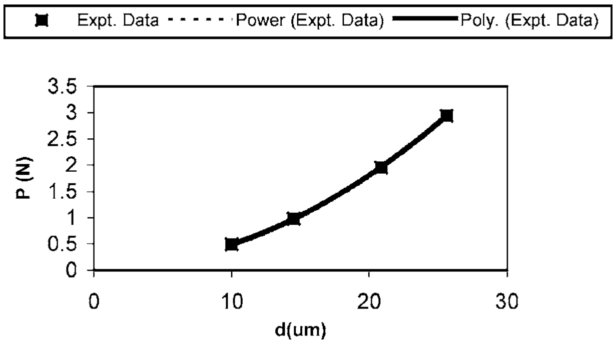
$$H_T = 1.8544 \times 0.0049 \frac{N}{\mu\text{m}^2} = 9.08\ \text{GPa}.$$

(b) For AlCoNi decagonal quasicrystal [7]

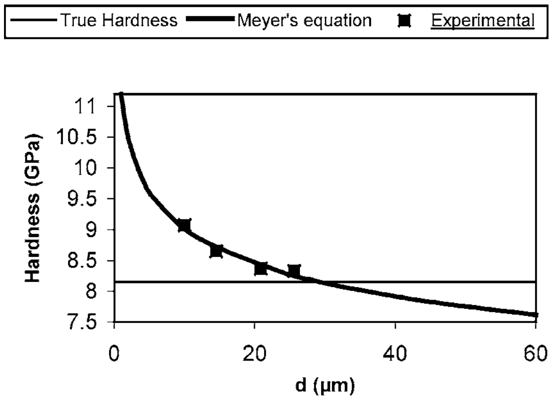
$$\text{Meyer's law: } P = 0.0061 d^{1.9059};$$

$$\text{Energy Model: } P = 0.058 - 0.0011d + 0.0044d^2$$

$$H_T = 1.8544 \times 0.0044 \frac{N}{\mu\text{m}^2} = 8.16\ \text{GPa}.$$

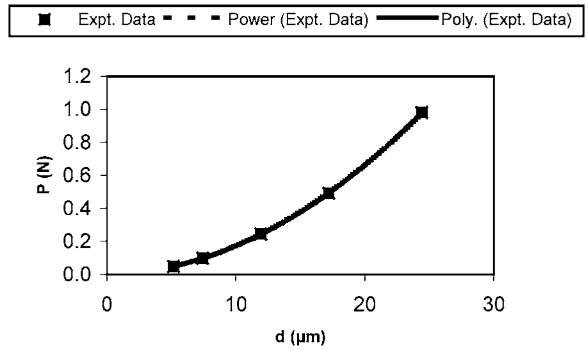


(a)

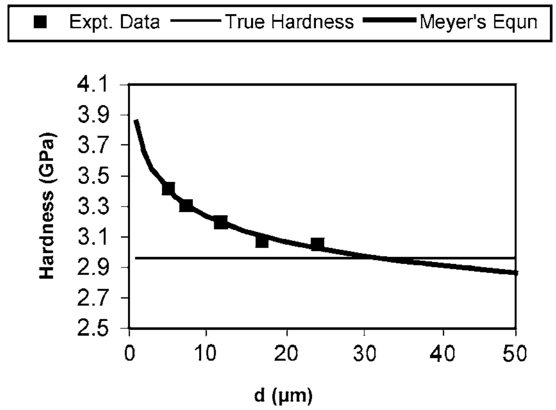


(b)

Figure 2 (a) Plot of the variation of load versus indentation diagonal obtained from Vickers microindentation experiment [7] of the AlCoNi decagonal quasicrystalline phase. (b) The experimental hardness data, the hardness curve from Meyer's Equation 6b, and the True hardness line obtained from energy balance model, are plotted against the indentation diagonal.



(a)



(b)

Figure 3 (a) Plot of the variation of load versus indentation diagonal obtained from Vickers microindentation experiment [16] of the $Mg_{32}(AlZn)_{49}$ intermetallic phase. (b) The experimental hardness data, the hardness curve from Meyer's Equation 6b, and the True hardness line obtained from energy balance model, are plotted against the indentation diagonal.

(c) For Mg_{32} $(AlZn)_{49}$ intermetallic compound [16]

Meyer's law: $P = 0.0021d^{1.9243}$;

Energy Model: $P = 0.0021 + 0.0011d + 0.0016d^2$

$$H_T = 1.8544 \times 0.0016\frac{N}{\mu m^2} = 2.97 \text{ GPa}.$$

Now using the Equation 10 d^* are determined to be 70, 27, and 36 μm for AlCoCu, AlNiCo quasicrystals and $Mg_{32}(AlZn)_{49}$ phase respectively. This appears to be reasonable and consistent with the trend of the hardness plot with the indentation size, which can be seen in Figs 1b, 2b, and 3b. The intersection between the Meyer's curve of hardness and the true hardness line can be seen clearly. The true hardness for AlCoCu, AlCoNi and $Mg_{32}(AlZn)_{49}$ phases was found to be 9.01, 8.16, and 2.97 GPa, respectively, which also seem to be consistent. The present analysis lends a strong support to the proposed approach for finding out the critical indentation diagonal by involving both the Meyer's power law equation and energy balance model.

Thus, it can be concluded that the normalized Meyer's equation proposed here can give rise to a better understanding of the Meyer's constant (K_m) and it's exponent (n). These parameters combined with the coefficient of the energy balance model can predict the critical indentation size after which ISE ceases to exist. Following this approach, the true hardness and the critical indentation size were found for AlCoCu, AlCoNi quasicrystalline, and $Mg_{32}(AlZn)_{49}$ crystalline phases.

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