

Important Consequences of the Exponent 3/2 for Pyramidal/Conical Indentations-New Definitions of Physical Hardness and Modulus

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Abstract

The now physically founded exponent 3/2 that governs the relation of normal force to depth^{3/2} in conical/pyramidal indentation is a physically founded ($F_N = k h^{3/2}$). Strictly linear plots obtain non-iterated penetration resistance k (mN/ $\mu\text{m}^{3/2}$) as slope, initial effects (including tip rounding), adhesion energy, and phase transitions with their transformation energy and activation energy. The reason for the failing of the Sneddon theory, claiming wrong exponent 2 (as do ABAQUS or ANSYS finite element simulations) is their neglect of long-range effects by shearing. Previous undue trials to rationalize the non-occurrence of exponent 2 are polynomial fittings and "best or variable exponent" iterations for curve fittings that lose all unique information from the loading curve. Also ISO 14577 unloading hardness H_{ISO} and reduced elastic modulus $E_{r\text{-ISO}}$ lack physical reality. They are redefined to physical dimensions as new indentation parameters H_{phys} and $E_{r\text{phys}}$. For the first time physically sound indentation hardness H_{phys} is obtained without iterations solely from loading curves. Also all mechanical indentation parameters relying on Sneddon's exponent 2 are unphysical. They require redefinition with new dimensions. This applies also to visco-elastic-plastic parameters in a recent NIST tutorial. The present ISO-standards create dilemma with physics. But the risk from using wrong mechanical parameters against physics is dangerous, subject to change.

Keywords: Adhesion energy; Composites compatibility; First energy law violation; Hardness and modulus definition; Indentation exponent; Physical consequences; Penetration resistance; Material's failure risk; Undue ISO-standards; Undue tutorial

Abbreviations

- AFM: Atomic Force Microscopy;
- CFG: Common Fine Grain;
- FE: Finite Element;
- ISO: International Standardization Organization;
- JKR: Johnson, Kendall, and Roberts technique;
- NIST: National Institute of Standardization and Technology;
- PEEK: Polyetheretherketone;
- PMMA: Polymethylmethacrylate;
- POM: Polyoxymethylene;
- UFG: Ultra-fine grain

Introduction

Instrumental nano-, micro-, and macro-indentations are still primarily standardized with diamond Berkovich indenters according to ISO 14577. These standards rest on diverging mathematical deductions of Love [1] and Sneddon [2] claiming proportionality of the applied normal force F_N (they called it P) and h^2 for the loading curves (h is penetration depth) for all kinds of (pseudo)conical indenters. This found widespread belief in publications and textbooks, but experimental loading curves do not show such relation. Several iterative "excuses" for this inconsistency were proposed, and finite element (FE) simulations continue to converge with exponent 2 on h . Claims that these would reproduce experimental loading curves are incorrect [3,4], the published experimental curves analyze with exponent 3/2. In that situation ISO 14577 concentrated on the iterative analysis of the unloading curve with freely iterated exponent on h (between 1 and 3) for gaining values of indentation hardness H_{ISO} and reduced elastic modulus $E_{r\text{-ISO}}$. Such

iterations are with respect to standard materials and projected area A_{hc} . Analyses (rather than fitting) of F_N versus $h^{3/2}$ plots of published loading curves in the literature (and of own ones) starting from 2000 by the present author [5] validated the exponent 3/2 by linear regression with excellent correlation coefficients of at least $r > 0.999$ and in less noisy cases $r > 0.9999$ for the materials penetration resistance k (mN/ $\mu\text{m}^{3/2}$). Nevertheless, this met with severe difficulties from anonymous referees for being supported and published, as these claimed to consistently find "exponent 2" on h . But analysis for exponent 2 or 3/2 is a matter of some minutes with Excel, provided correct experimentation. Liability facts and unexpected applications with precise calculation were hardly appreciated. Only the correct analysis (Eq. 1) with excellent linear regression reveals surface effects (including tip rounding), influence of tip angle and radius on k , gradients, mechanical pretreatment, alternating layers, elbows, nanopores, phase transitions under load, transition energies, activation energies, and correct adhesion energies, all by simple mathematics without iterations (Eq. 1) [4,6-9].

$$F_N = k h^{3/2} \quad (1)$$

The constant k (mN/ $\mu\text{m}^{3/2}$) is the penetration resistance, a materials property that is obtained with the highest precision in the (nano) indentation experiment, rather than multi-iterated hardness, reduced modulus, etc. After the recent physical foundation of the exponent 3/2, giving the explanation why it must be so, by considering both the simultaneous volume-formation and the thereby created total pressure

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with elementary mathematics [10], the physical law (1) is additionally enforced beyond any doubt and cannot be denied any more. We must herewith point out the necessity of using this new state of the art for removal of the dilemma between ISO 14577 and physics (for undue NIST tutorial from 2009) for all mechanical parameters that rely on h^2 . They must be re-defined, and all the corresponding mechanical data require correction. Also the critics of three different working groups on the exponent 3/2 on h [11] (before its physical deduction) deserves retraction: the self-similarity of conical/pyramidal indenters is by no means a "straightforward proof" for the unsupported exponent 2 on h , but it violates the basic energy conservation principle.

Materials and Methods

A fully calibrated Hysitron Inc. TriboScope® Nanomechanical Test Instrument with a two-dimensional transducer and leveling device in load control mode was used for the author's nanoindentations after due calibration, including instrument compliance. The radii of the cube corner (55 nm) and Berkovich (110 nm) diamond indenters were directly measured by AFM in tapping mode. Three-dimensional microscopic inspection of the indenter tips secured smooth side faces of the diamonds for at least 2 μm from the (not resolved) apex. The samples were glued to magnetically hold plates and leveled at slopes of $\pm 1^\circ$ in x and y directions under AFM control with disabled plain-fit, and loading times were 10–30 s for 400–500 or 3000 data pairs [4]. The whole data set of the loading curve was used for analysis, using Excel®. Most analyses were however with published loading curves from the literature, as rapid sketches with pencil, paper, and calculator (10-20 data pairs), but for linear regressions always by digitization to give 50-70 almost uniformly arranged data pairs using the Plot Digitizer 2.5.1 program (www.Softpedia.com), unless complete original data sets could be obtained from the scientists. The precise kink positions were obtained by equating the linear branches before and after the phase change, and precise axis cuts from the regression lines. It was tried to cover all different materials types, all different indentation modes, equipments, response mechanisms, depth ranges, penetration resistance sizes, from numerous authors from all around the globe. Only the experimental curves are relevant, not the simulated ones.

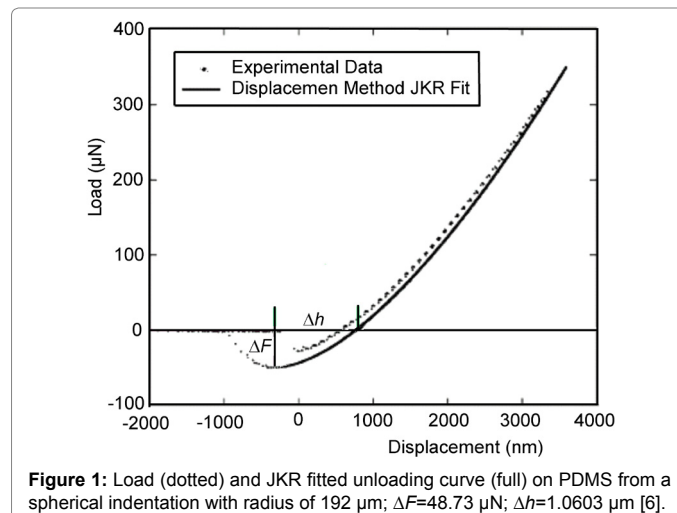
Results and Discussion

Information loss by finite element simulations, beliefs, polynomial fittings, and exponent iterations

Finite element simulations of loading curves (ABAQUS or ANSYS, etc.) consistently converge with the exponent 2 on h (e.g. [3]). There is thus never match with experimental results. However, there are claims that microindentations would require "exponent 2". For example, Oliver and Pharr [12] depicted in 1992 deep non-discussed Berkovich micro-indentation loading curves of soda lime glass, sapphire, fused quartz, and α -quartz (001) up to 120 mN load, for obtaining unloading curves for hardness and elastic modulus iterations. But the former were not focal for that paper on unloading curves. All of these "loading curves" analyze with the impossible F_N versus h^2 relation, but experimental curves from various authors (including WC Oliver) go with $h^{3/2}$ (including those with phase change kink), as for example analyzed in [4,13]. I apologize for having believed in their validity in 2005/6 [14]. There is, however, no exponential differences between nano- and micro- or macro-indentations (as long as these proceed properly with smooth tips and without cracks). All of these loading curves obey Eq. (1) [4,6,8]. It is thus not clear why the loading curves in [12] analyze with the (now disproved) Love/Sneddon exponent. Importantly, only

the correct exponent could reveal the phase change of the standard fused quartz for H and E_r iterations at about 2.5 mN load [4,13].

The polynomial fitting ($F_N = c_0 h^2 + c_1 h + c_2$) for the total loading curve [15] has been widely applauded. But it provides no information about initial effects, gradients, or phase transformations at all, and polynomial fittings are unreliable in view of linear regressions. Furthermore, iterated parameters c_0 and c_1 are often used to calculate exceedingly large "effective tip radii" up to 3.3 μm (for example for a Vickers with 68° semi-angle Θ that is close to the one of Berkovich at 65.3°), depending on the yield-strength/modulus ratio [15]. However, blunt Berkovich tip radii range from 150 to 300 nm. Such incredible polynomial fitting parameters, are being used as an excuse for not obtaining the believed exponent 2 at the expense of linear regression with the physical exponent 3/2. Importantly, properly executed experimental loading curves are required for exponential analyses. This implies indenters with flat side-faces, vertical (not tilted) indenting onto homogeneous materials with plain surfaces that must not be influenced by nearby impressions, or too close sample edges or sample borderlines, in order to avoid strange results with spurious too large exponents up to >1.5 or even >2 . Unfortunately, leveling equipment for skew surfaces (with AFM precision check) often lack in commercial nanoindentation instruments. Nevertheless, measurements with blunt Berkovich ($R \approx 300$ nm) giving unusually long initial effects were tried to "discredit" the exponent 3/2 with the F_N versus $h^{3/2}$ plot of fused quartz in Figure 1 [16]. However, this plot confuses the 3 initial-effect points with the not considered straight line through the points # 4–17 at the actual kink position where the steeper second linear branch starts. Rather, the authors absurdly intersect a line through points # 1–3 with the extrapolated second straight line from point17 onwards. Such intersection is far away from the plot. The false claim is then made that "Kaupp" would also have intersected with initial effect points at his analyses of the same material in [4,13,14,17]. However, Kaupp has always excluded initial effects at his regressions, and he provided obvious reasons for their occurrence. The actual kink (k_1 and k_2 lines) is at about 3.4 mN and 195 nm. This is not too far away from the values for the known sharp Berkovich indents (2-2.5 mN and 120-160 nm) [4,13,14,17] at very minor initial effects. The experimental data printed curves of [16] are therefore *supporting* but not at all "discrediting" the exponent of Eq. (1), if considering the unusually extended initial effect range (axis cut of the k_1 line at about -1.2 mN; not drawn in [16]) at this indentation. Nevertheless, the authors in [16] deny their obvious support of $h^{3/2}$. Rather they undertook exponent fittings with $F = kh^n$



(not italicized for distinction from physical k values) for varying depths onto fused quartz for proposing varying depth/exponent and depth/ k relations. They refrain from indicating the dimensions of the so obtained variable proportionality constants. The published data sequence for different final depths onto fused quartz is as follows [16] (only the dimensions are now added): Up to 40 nm depth, $n=1.5$, $k=0.99411$ mN/nm^{1.5}; upto 60 nm, $n=1.64533$, $k=0.61897$ mN/nm^{1.64533}; upto 80 nm, $n=1.75285$, $k=0.41377$ mN/nm^{1.75285}; upto 300 nm, $n=1.82723$, $k=3.00003E-4$ mN/nm^{1.82723} for the overall curve [16]. It does not help to compare with FE simulations including further parameters. All of that is physically unsound and totally meaningless, but obviously not recognized by the authors, referees, and editors [16]. Clearly, one was not willing to recognize specific properties of materials under loading stress and strangely strived for concurring with the disproved Sneddon theory. The exponent on h is with mathematical precision 3/2 and the dimension of k is (force/length^{3/2}) [10]. Minimal deviations are experimental errors. Initial effects (including tip rounding contribution) are quite common, and phase transitions upon indentation loads prior to macroscopic cracking are frequent. These are important properties!

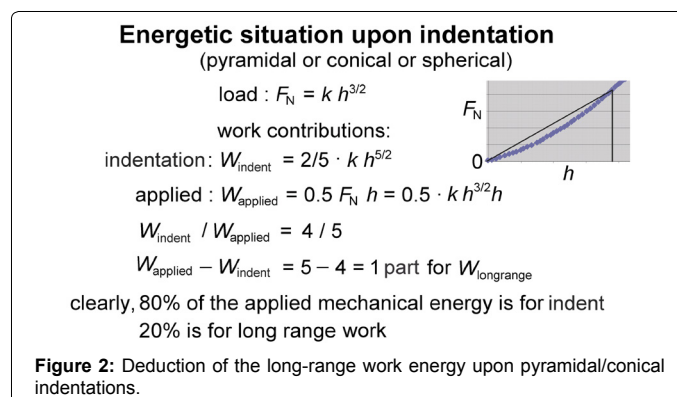
Different iteration induced flaws, provides the JKR (Johnson, Kendall, and Roberts) treatment of adhesion forces, even though these iterations start with the Hertzian exponent 3/2 for spherical tips or rounded AFM cantilevers. However, based on the penetration resistance k the +166% error of the JKR-adhesion work is easily revealed both from indentations and AFM force curves. This has first been described in [6] and is another important application of Eq. (1). Both curves (loading and iterated unloading) in Figure 1 strictly follow Eq. 1 as well. Thus the full gained adhesion work (determined as pulloff work) is calculated by the triangle area, as described by ΔF and Δh , of the unloading curve giving the absolute value for $W_{\text{pulloff}}=0.5 \Delta F \Delta h=25.8342$ $\mu\text{N}/\mu\text{m}$. The published JKR treatment reported the $\Delta\gamma$ -value (that is called a basic JKR-formula) as $\Delta\gamma=2 F_{\text{adhesion}}(h)/3\pi R(h)$, where $R=192$ μm is "the radius of curvature of the tip, and the reported $\Delta\gamma$ is 0.05389 $\mu\text{N}/\mu\text{m}$, which is dimensionally a force constant, but it can also be interpreted as work over area by extension of the formula, as indicated by the bracketed h . By division of the absolute W_{pulloff} value from our not iterating ENERGY technique [6] by the calotte surface of the spherical tip ($2 \pi R h$) one obtains $W_{\text{adh}}/\text{area}=0.5 F_{\text{adhesion}} h/2\pi R$ $h=0.02020$ $\mu\text{N}/\mu\text{m}$, now with the same dimension for comparison with the JKR $\Delta\gamma$. We immediately see from the quoted $\Delta\gamma$ formula that JKR divides 4 times the work over 1.5 times the area, instead of ENERGY technique's correct work/area. The mathematically exact error of JKR calculates therefore from the wrong numerals 2/3 in its "basic formula" instead of 0.5/2. The ratio 2/3 over 0.5/2 is 8/3=2.6667. This corresponds to a JKR error for $\Delta\gamma$ of $(8-3)100/3=+166\%$ that is nicely confirmed by the numerical data: $0.05389/0.02020=2.66716$. This huge JKR error is particularly detrimental, as it also occurs (with the same 166% error) in AFM force-measurements (a typical example is presented in [6]) and that these $\Delta\gamma$ -values are used for the "determination" of reduced elastic modulus values according to the JKR formula $E_r=9\pi R^2 \Delta\gamma / 2a_0^3$ that are thousand-fold "determined", used, and tabulated, particularly with soft medical/biological samples. There must be correction of all the tabulated elastic moduli from JKR-adhesion work.

Particularly strange are suggestions to deny the universal exponent 3/2 on h , and the unexpected practical applications of Eq. (1), but to base the exponential analysis on FE simulations [11]. The authors from the three research groups do not separate out the initial surface effects and deny often occurring phase transformations, obviously because the search for them is impossible with FE simulations. It is unscientific to use fitted data or FE-simulated curves that converge to h^2 , and to

recalculate these for $h^{3/2}$ with the aim to discredit the experimental (now physically founded [10]) exponent 3/2, because such treatment inevitably gives bent curves. Such a "treated" curve was used for drawing tangents at the start and the end in Figure 2 of [11] that intersect far away from the plot, for designing a false discrediting term called "Double $P-h^{3/2}$ fit after Kaupp et al." [11]. However, Kaupp et al. do not fit treated data but are analyzing experimental loading curves according to the physically deduced universal Eq. (1) [10] to uncover individual properties (e.g. phase change yes or no) that are wiped out by data fittings or simulations as in [11,16]. It is unclear, where the data of [11] in opposition to physics [10] and to the published ones came from, and who did the calculations for fused quartz up to 300 mN load on what assumptions. The polynomial or FE correlations (Table 1) [11] are not helpful (for example phase changes are unavoidable for the partially crystallized POM and PEEK thermoplastics or compacted Al). Figure 2 in [11] report either very different exponents (1.6 to 2.15 between 200 and 1300 nm depth) in different loading ranges, or a "constant exponent 2" for the linear P versus h^2 plot, respectively: a very severe discrepancy! And the above reference [16] is invoked with its unintentional support of Eq. (1) and the phase transition of fused quartz. Furthermore, earlier experimental loading curves of the author K Durst et al. [4] (e.g. spruce or UFG and CFG Fe), as analyzed in Figure 2, precisely follow Eq. (1) with sharp kinks at 0.53, 0.87, 27.5, and 40.9 mN load.

Penetration resistance reveals phase transformations

When within the loading range of the linear plot a sudden sharp kink discontinuity occurs, this is the onset of a phase change under load (numerous images for fused quartz and all types of materials [4,13,14,17]). This is one of the reasons for errors that have not been addressed with H_{ISO} and $E_{r\text{-ISO}}$ determinations. Only properly analyzed loading curves (Eq. 1) detect or exclude (Table 1) and plots in [4] phase changes of all kinds (not only structural transitions), but neither exponent fittings nor present FE simulations can do so. Phase changes occur with many materials already in the nanoindentation range. Only rarely and exceptionally were phase transitions concluded from "elbows" in unloading curves, but then without any transition-onset information. A widely studied example, also with more advanced techniques, is silicon [7]. Original material is characterized by the penetration resistance k_1 before the kink in linearized loading curves. After the kink the k_2 -value is obtained for transformed material in a matrix of the original one [4]. It provides an important bargain when both k_1 and k_2 (mN/ $\mu\text{m}^{3/2}$) are known: The transition energy [6], and temperature dependent also the activation energy of the transition are revealed [7]. When the onset of phase changes is not uncovered, there is often a risk that such transition-onset has occurred before the applied



load. Clearly, not detected phase changes bear a high risk for materials' failure upon aging under load and heat stress, when the mechanical load at the given temperature surpasses the transformation onset, for example with alloys, or ceramics, and other composites. Such failure upon impact can grow-up to disasters. Already that urges the correction of the corresponding ISO-standards as soon as possible.

Hardness and modulus

ISO 14577 uses the unloading curve for iteratively obtaining the hardness H_{ISO} with respect to standard materials (mostly fused quartz, or Al) with freely iterated exponent (m: 1–3). These iterations converge with respect to the standard material of "known" mechanical properties. However, the standard materials have in most cases different surface effects, and both undergo widely ignored phase transitions after their onset discontinuity (fused quartz in the nano-, aluminum in the micro-indentation region) [4,13].

The main objection of referees against Eq. (1) was the definition of the universal hardness (Martens hardness) as normal force over projected area $A_{proj} = \pi h^2 \tan^2 \alpha$ (α = effective cone angle of 70.2996°) (Eq. 2) with unit $mN/\mu m^2 = GPa$. This covers the volume of a tube $A_{proj} h_{max}$. But Eq. (2) is undue, as it implies the false " $F_N \propto h^2$ " as in Eq. (3).

$$H_{universal} = F_{Nmax} / A_{proj} \quad (2)$$

$$F_{Nmax} = \pi R^2 H_{universal} \text{ and } R/h = \tan \alpha \text{ would give } F_{Nmax} = \pi h^2 \tan^2 H_{universal} \quad (3)$$

ISO uses normal force over contact area A_{hc} (effective πR^2) as opposed to the cone area ($2\pi R^2/\tan \alpha$). Thus the covered volume is meant as the tube $A_{hc} h_{max}$ or $A_{hc} h_c$. But physics [6] beats definitions. The "contact height" h_c is defined with reference to the standard at peak load Eq. (4), where $\epsilon=0.75$ is an accepted but also debated dimensionless geometric factor, and S is unloading stiffness ($mN/\mu m$), the unloading slope at peak load. The iterative determination of H_{ISO} fits to the generally unrelated fused quartz that experiences some sink-in ($h_s = h_{max} - h_c$, depending on the indenter) for A_{hc} at peak load. The iteration requires $F_N = B(h_{max} - h_c)^m$ where B , h_{final} , and exponent m (between 1 and 3) are freely iterated for the fitting down to 20% or eventually 50% of F_{Nmax} , which is also troublesome indeed [14,17]. S at peak load is then obtained by differentiation [$S = Bm(h_{max} - h_c)^{(m-1)}$] to obtain h_c , for the calculation of $A_{hc} = 3^{3/2} h_c^2 \tan^2 \theta = 24.56 h_c^2$ as a first guess for Berkovich ($\theta = 65.3^\circ$) indentations. H_{ISO} is then iterated according to Eq. (5). Thus, H_{ISO} (Eq. 5) also implies an $F_N \propto h_c^2$ relation, which is against physics as is Eq. (3), both violating the physically founded Eq. (1) [10] that is also valid for F_{Nmax} , of course. Furthermore, any sink-in or pile-up is part of the energy requiring long-range effects.

$$h_c = h_{max} - \epsilon F_{Nmax} / S \quad (4)$$

$$H_{ISO} = F_{Nmax} / A_{hc} = F_{Nmax} / (24.56 h_c^2 + C_1 h_c + C_2 h_c^{1/2} + C_3 h_c^{1/4} + \dots + C_8 h_c^{1/128}) \text{ [mN}/\mu m^2] \quad (5)$$

Rather strange trials to "save" h^2 and thus A_{hc} for the hardness iterations are still invoking the trivial "self-similarity" of conical or pyramidal indenters for claiming " $A_c \propto h^2$ " and " $H = P/A_c$ " (P stands for F_N) [11]. Such assertion is surprising since it is amply known and discussed, that the shear-force from the cone or pyramid produces far-reaching phenomena to the sides and below the defined tube volume (these include sink-in or pile-up, plastic deformation, elastic stress, rosettes, shear-bands, and so on) [6]. Such long-range processes convert or store energy that is lost for the impression. The denial of such loss inexcusably violates the basic first energy law (applied work = produced work/energy): Equation (1) and simple algebra deduce

that precisely 20% of the applied work (for all materials) is used for the sum of long-range effects and lost for the indentation depth [6-10]. The energy loss for the penetration as deduced in [6], is summarized in the basic Figure 2. The experimental loading curve follows $F_N = k h^{3/2}$ (Eq. 1) in all cases, also the spherical ones and the indentation work is obtained by integration to give $W_{indent} = 0.4 k h^{5/2}$. This compares with the linearly applied work (area of the triangle up to the chosen depth h), which amounts to $W_{applied} = 0.5 F_N h = 0.5 k h^{5/2}$ by substitution of F_N . It is thus arithmetically clear, that precisely 20% of the applied work is lost for the indentation work due to the sum of long-range work contributions [6]. This is therefore valid for all materials and mechanisms (e.g. elastic and plastic deformations, material's migrations, sink-in, pile-up, viscous flow) for all pyramidal/conical properly executed indentations, when detectable initial surface effects (that also contain tip rounding effects) are corrected for. In the case of sharply detected (kink in the plot of Eq. 1) phase change onset (endo- or exo-thermic), a second energetic term that includes the transition energy is calculated accordingly [6]. Both W_{indent} and $W_{longrange}$ require their part from the normally applied force F_N , mathematically at an always constant ratio [6]. Thus, Eq. (2) (universal hardness) and (5) (ISO-hardness) violate not only the physically founded indentation law (1) but both hardness definitions also violate the first energy law, because they do not take into account that part of F_{Nmax} that is lost for 20% of the work. They have been doing so for half a century.

In order to stay within the energy law it is not necessary to care for a definition of ensuing "lengths" for the long-range work. The change of the ISO 14577 hardness H_{ISO} has to occur in such a way that the $F_N \propto h^{3/2}$ relation (Eq. 1) and thus the first energy law are not violated. The same is necessary for $H_{universal}$. The viable redefinition of $H_{universal}$ or the previous ISO-hardness, is thus by multiplication of $F_{Nmax} / A_{projected}$ (or F_{Nmax} / A_{hc}) with $h_{max}^{1/2}$ (Eq. 6). Since sink-in and pile-up effects do not influence the exponent [10], the H_{phys} -values (that do not longer depend on a standard) do not require A_{hc} , and the universal $F_N \propto h^{3/2}$ relation and the first energy law are obeyed by taking into account the long-range losses (long-range energy that requires part of the applied force) (Eq. 7). The dimension of physical indentation hardness H_{phys} has thus the units $mN/\mu m^{3/2} = GPa \mu m^{1/2}$, the same as the dimension of penetration resistance k in Eq. (1). By substitution of F_{Nmax} in (7) with (1) one obtains therefore a precise and simple way to obtain the physical indentation hardness H_{phys} directly from conical/pyramidal indenters' loading curves (Eq. 8). The $h_{max}^{3/2}$ factors cancel out for H_{phys} . It follows from Eq. (1) (that is also valid for F_{Nmax}) that only the loading curve is

$$H_{phys} = F_{Nmax} h_{max}^{1/2} / A_{proj} \quad (6)$$

$$F_{Nmax} = \pi R^2 H_{phys} / h_{max}^{1/2} \text{ and } R/h_{max} = \tan \alpha \text{ gives } F_{Nmax} = \pi h_{max}^{3/2} \tan^2 H_{phys} \quad (7)$$

$$H_{phys} = k / \pi \tan^2 \alpha \text{ (mN}/\mu m^{3/2}) \quad (8)$$

required for H_{phys} (Eq. 8), with the dimension of the penetration resistance k , opens an easy and simple way to obtain the physically sound hardness H_{phys} ($mN/\mu m^{3/2}$), without iterations, only from the loading curve with the unbeatable penetration resistance k_i (before the kink) by linear regression. The tip rounding initial effect is not part of the linear regression of the penetration resistance (but it plays a role for adjustments between different pyramids/cones [4,18]). For the first time, this new technique controls the final load below any phase transition onset. The precise technique makes obsolete the iterative determination of a "contact area", and undue experiments (e.g. tilted, too tight with others, etc.) are easily detected. Unfortunately, conversions of previous H_{ISO} into H_{phys} values are not easy due to the various iterations within

the ISO-treatment, but $H_{\text{universal}}$ does not have iterations due to totally different standard. The h_{max} values would however require the loading curves or original data. The correction of indentation $H_{\text{universal}}$ (GPa) into H_{phys} (GPa $\mu\text{m}^{1/2}$) works by

a) isolation of h_{max}^2 :

$H_{\text{universal}} \pi \tan^2 \alpha / F_{\text{Nmax}} = 1/h_{\text{max}}^2$ (mN/ μm^2 ; non physical); H_{ISO} known, F_{Nmax} must be known;

b) calculation for $h_{\text{max}}^{3/2}$:

$H_{\text{phys}} \pi \tan^2 \alpha / F_{\text{Nmax}} = 1/h_{\text{max}}^{3/2}$ (mN/ $\mu\text{m}^{3/2}$; physical); H_{phys} can be calculated, when F_{Nmax} is known.

The results are exemplified in Table 1 from a paper [3] that published both experimental loading curve and finite element simulation loading curves far below of a phase change onset. One remarks considerable differences between H_{ISO} (from unloading curve with excessive iterations of aluminum on silicon for fit with the standard) and $H_{\text{universal}}$ (invoking h^2) without correction. On the other hand, it is clear that the corrected $H_{\text{universal}} = H_{\text{phys}}$ is very different from the uncorrected $H_{\text{universal}}$ numerically (h_{max} was 0.250 μm , giving the factor 2 for $h_{\text{universal}}/h_{\text{phys}}$) and, of course dimensionally. Importantly, it is easiest to correct the finite element H -value, because the unphysical exponent 2 cancels out by the correction with $h_{\text{max}}^{1/2}$, and H_{phys} and simulated H_{corr} are quite similar. However such good correspondence is only possible when absence of phase change onset is experimentally secured at h_{max} , which finite element simulation cannot predict or exclude. The necessary k and k' values must still be determined from loading curves (Eq.1 or analogous for simulated k' with different dimension).

Similar to H_{ISO} , the definition of the modulus $E_{\text{r-ISO}}$ contains A_{hc} , which does not comply with the long-range effects at peak load, against physics Eq. (9) [8]. A quantitative connection to the penetration resistance k of the loading curve is however lost, because the unloading slope is needed for S (mN/ μm). Also here, peak load must be below any kink load in the linearized loading curve (Eq. 1), in order to study the pristine material. It follows for both reasons that $E_{\text{r-ISO}}$ values of indentations (Eq. 9) are not appropriate, because they also violate physics by neglecting the energy consuming shearing effects. As in the case of hardness, the correction factor with respect to absolute values must be $h_{\text{max}}^{1/2}$, when using A_{proj} (not the iterated A_{hc}) for obtaining the physically correct dimension. This is done for $E_{\text{r-phys}}$ with its substitutions as in Eq. (10), from which Eq. (11) follows arithmetically. The physical dimension of indentation $E_{\text{r-phys}}$ is thus (mN/ $\mu\text{m}^{7/4}$) or (GPa $\mu\text{m}^{1/4}$). Future stiffness determinations for the physical modulus from indentations require proper modification.

$$E_{\text{r-ISO}} = 1/2 S / 2 A_{\text{hc}}^{1/2} \quad (9)$$

$$E_{\text{r-phys}}^2 = h_{\text{max}}^{1/2} \pi S^2 / 4 A_{\text{proj}} = h_{\text{max}}^{1/2} \pi S^2 / 4 \pi R^2 = h_{\text{max}}^{1/2} S^2 / 4 h_{\text{max}}^2 \tan^2 \alpha = S^2 / 4 h_{\text{max}}^{3/2} \tan^2 \alpha \quad (10)$$

$$E_{\text{r-phys}} = S / 2 h_{\text{max}}^{7/4} \tan \alpha \quad (11)$$

The correction of indentation $E_{\text{r-ISO}}$ (GPa) into $E_{\text{r-phys}}$ (GPa $\mu\text{m}^{1/4}$) works by

(a) isolation of h_{max}^2 :

$E_{\text{r-ISO}}^2 4 \tan^2 \alpha / S^2 = 1/h_{\text{max}}^2$ ($E_{\text{r-ISO}}$: mN/ μm^2 ; is non physical); $E_{\text{r-ISO}}$ is known; h_{max} and reliable S before phase change onset must be known;

(b) calculation for $h_{\text{max}}^{3/2}$:

$E_{\text{r-phys}}^2 4 \tan^2 \alpha / S^2 = 1/h_{\text{max}}^{3/2}$ ($E_{\text{r-phys}}$: mN/ $\mu\text{m}^{7/4}$ is physical); $E_{\text{r-phys}}$ can be calculated, when h_{max} and reliable S are known.

The application of non-physical H_{ISO} and non-physical $E_{\text{r-ISO}}$ as well as from these derived further mechanical parameters in theoretical and practical mechanics, bears high risks for materials' performance and must be stopped. Tabulated materials' properties must become reliable for centuries. Therefore, the most precise and undeniable penetration resistance k , H_{phys} and $E_{\text{r-phys}}$ should be urgently used for the characterization of materials and the compatibility of different materials, including solder etc. [8,9].

Undue tutorial parameters

All "Sneddon theory"-derived mechanical indentation parameters rely directly or indirectly on the presumed incorrect " $F_N \propto h^2$ " relation. This is exemplary demonstrated with some of the incorrect parameters that ensue. For example, NIST authors [19] published a tutorial in 2009, as based on their earlier publication [20], with the erroneous deduction of six "viscous-elastic-plastic mechanical parameters". The authors used their loading-rate dependent Berkovich indentations onto PMMA and claimed that all of their published curves [19,20] would obey the Sneddon exponent 2 on h . But NIST could have easily found out that their loading curves of PMMA with a sharp Berkovich indenter fantastically support the universal F_N versus $h^{3/2}$ plots (Eq. (1)) with excellent straight lines for all of their loading rates from zero to their very deep penetrations (down to maximal depth of 6.5 μm), within less than 10 min, and with excellent linear regression. So NIST missed the validity of exponent 3/2 instead of 2. Rather, starting with the non-supported equations " $F_{\text{Np}} = \alpha_1 H h_p^{2/3}$ " and " $F_{\text{Ne}} = \alpha_2 E_r h_e^{2/3}$ " (sub-p for plastic, sub-e for elastic) and after various steps (with inclusion of a "quadratic viscous element") they defined thus six incorrect mechanical parameters:

" $y = (\alpha_1 H / \alpha_2 E_r)^{1/2}$ " as "indentation plastic yield resistance",

" $d = 3\tau/2t_r$ " as "indentation viscous flow resistance" (with t_r as rise time), and the double logarithmic plot of y versus d was termed "indentation behavior map",

" $1/e = 1 + 1/y + 1/d^3$ " (with e as "elastic fraction"),

" $H = F_{\text{Nmax}} y^2 / h_{\text{max}}^2 \alpha_1 e^{2/3}$ ", and " $E_r = F_{\text{Nmax}} / h_{\text{max}}^2 \alpha_2 e^{2/3}$ ".

Technique	h_{max}^n	k or k' (a)	Basis	Hardness only from the loading curve (not H_{ISO})
Experimental with $h_{\text{max}}^{1/2}$ factor	$h_{\text{max}}^{3/2}$	$k = 7.4425$ (mN/ $\mu\text{m}^{3/2}$)	$F_N = kh^{3/2}$	$H_{\text{phys}} = k/\pi \tan^2 \alpha = 0.30373$ (mN/ $\mu\text{m}^{3/2}$) independent on F_N and h_{max} (before phase change)
Experimental no correction	h_{max}^2	$k = 7.4425$ (mN/ μm^2)	$F_N = kh^2$ wrongly	$H_{\text{ISO}} = 0.761$; $H_{\text{univ}} = k/\pi \tan^2 \alpha = 0.60745$ (mN/ μm^2) $k + h_{\text{max}}^{1/2}$ are needed for correction (value + dimension)
Finite element no correction	h_{max}^2	$k' = 4.7433$ (mN/ μm^2)	$F_N = k'h^2$	$H_{\text{uncorr}} = k'/\pi \tan^2 \alpha = 0.60167$ (mN/ μm^2) false dimension, multiply with $h_{\text{max}}^{1/2}$ when available
Finite element with $h_{\text{max}}^{1/2}$ factor	h_{max}^2	$k' = 4.7433$ (mN/ μm^2)	$F_N = k'h^2$	$H_{\text{corr}} = k'h_{\text{max}}^{1/2}/\pi \tan^2 \alpha = 0.30083$ (mN/ $\mu\text{m}^{3/2}$) (k' with dimension mN/ μm^2 , must be determined)

(a) simulated parameters are not italicized .

Table 1: Comparison of an experimental unloading curve H_{ISO} of Al on Si with loading curve H_{phys} and ANSYS finite element simulated corrected or uncorrected H-values from [3].

There is no experimental or physical basis for that and these papers from NIST (a prominent ISO member) require urgent retraction (after the successful physical deduction of Eq. (1) [10]). It is to be expected that a tutorial from representatives of the US-agency, which is responsive for standardizations with close to legal character, will be largely understood as a "state-of-the-art". But it is against physics. Therefore, an enormous risk has been arisen with this "tutorial" that has already been widely taught and used to produce and tabulate wrong data, with the potential of doing harm primarily to biology and medicine, but also to all further research on (nano) mechanics.

Furthermore, all the other textbook mechanical indentation parameters that directly or indirectly rely on h^2 require re-deduction, by starting with the physically correct exponent 3/2, and it has always to be considered that their dimensions will unavoidably change. The indentation experiment is now a quantitative technique on the basis of the new physics with the penetration resistance k , the inverse of which has been called penetrability [4]. The easily obtained penetration resistance, detects phase transition onset and conversion energy as well as activation energy, etc. and it provides detection of physically correct indentation hardness H_{phys} with correct dimension, all from the loading curves without iterations or simulations.

Conclusion

The recent physical foundation of the universal exponent 3/2 on pyramidal/conical indentations enforces appreciation of the abundant empiric results, and that has important consequences. Thus, the ISO 14577 indentation hardness H_{ISO} , the reduced elastic modulus $E_{\text{r-ISO}}$, and the three from deduced mechanical parameters must be urgently corrected in dimension and value, to provide the physically sound H_{phys} , $E_{\text{r-phys}}$, and there from deduced parameters. Their perhaps odd appearing dimensions are the peculiarity of indentations with applying both normal force and lateral force at the same time. The present ISO definitions rely on a wrongly proposed [1-2] " $F_N \propto h^2$ " relation and thus also on the undue reference to "projected area A_{hc} ". Its application does not consider the far-reaching shear-force effects outside the A_{hc} tube and is thus violating the basic first energy law. Physically sound hardness is now for the first time obtained from the loading curves without iterations or simulations by only using simple algebra, and H_{phys} is now a genuine physical parameter. Also all other mechanical parameters for pyramidal and conical indentation that rely directly or indirectly on disproved " h^2 " from the loading curves require redefinition and re-determination by using $h^{3/2}$ instead of " h^2 ". The almost universally published wrong mechanical parameters from indentations and AFM force curves constitute high risks that are often adopted and defended, subject to change. The large errors caused by the wrong exponent are exponential dependent, not proportional. Any non-appreciation of the physical exponent is at risk for the stability of incorrectly-calculated composite materials and solders, as for example implanted endo-prostheses (mechanical adjustments to the actual bones of the composite, "bone cements", alloys, composite ceramics, coatings, and inlays, adhesion energies etc.), or composed materials of daily life (for example longevity of turbines, cars, airplanes, medical implants, etc.). Wrong parameters against physics (values and dimensions) must be adjusted to avoid scratching and failure, when materials are under mechanical and thermal stress. It is the penetration resistance k (mN/ $\mu\text{m}^{3/2}$) of the components that must closely be adjusted to coincide for the applied force and temperature ranges. Of particular importance are the now easily recognized phase changes under load as detected by the onset of sharp kink discontinuities in the F_N versus $h^{3/2}$ plots that must always be considered. Different components have their transitions at

different pressure and different temperature onsets. Thus, their now also available transformation- and activation-energies require the capabilities of the penetration resistance k (mN/ $\mu\text{m}^{3/2}$). This should help in adjusting the components of mechanical and thermal stressed super alloys with their grains and domains that must be optimized, etc.

Textbooks must be rewritten, new dimensions of mechanical indentation parameters accepted. This is a tremendous task, because all mechanical parameter's dimensions become different. For spherical indentations $h^{3/2}$ is long iteratively used, but please do not use JKR-technique with 166% error. Thus, both the Hertzian-type ($h^{3/2}$) and Sneddon-type (h^2) analyses that are offered to choose from at some instrumental AFM-software require correction or cancellation. H_{ISO} , $E_{\text{r-ISO}}$, E_p and all mechanical parameters that are derived from h^2 exhibit huge systematic errors after very complicated data treatments with iterations, simulations, and approximations. Fortunately, the now available physics avoids iterative data-fittings and allows for precise algebraic data evaluation. Nobody in the field can reasonably continue proceeding against basic physics. The numerous unexpected possibilities with quantitative indentation loading (or AFM force-curves' scanning) must be pursued on the available easy and precise algebra. Further unexpected applications are expected by the measurement of penetration resistance, H_{phys} , $E_{\text{r-phys}}$, W_{adh} , and mechanical phase transformation onsets with energies of transformation and energies of activation.

ISO and its member NIST cannot abruptly but only considerably change their standards, because all academic and industrial players are trained with their non-physical formulas and thinking from the hard to repeat or realize high mathematics of [1-2] that did not consider the first energy law. Therefore, ISO/NIST should now release a caveat relating to their ISO 14577 standards, telling that new physical results (with proper citation of open access work [10]) is being processed for unexpected revision, as a first step. This would create relieve from dilemma of ISO against physics and exempt security engineers in industry and administration who are bound to ISO standards from the dilemma concerning liability questions when using either physical state of the art or non-physical standards. Worldwide ISO is slow in making the urgent change of their ISO 14577 standards. The clear wording is required to speed this process for a smooth non-chaotic change to physical standards for the sake of every days' security.

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