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# The ISO Standard 14577 for Mechanics Violates the First Energy Law and Denies Physical Dimensions

**Kaupp G***

*University of Oldenburg, Germany*

## Abstract

The basis of the quantitative conical/pyramidal (nano) indentation, without fittings, iterations, or simulations, is the physically founded $F_N=k\ h^{3/2}$ relation. The constant $k$ (penetration resistance, mN/μm$^{3/2}$) from linear plot with excellent regression discards initial surface effects, identifies important phase transformation onsets, conversion and activation energies, and reveals errors. The failing Sneddon theory of ISO with unphysical exponent 2 on $h$ lacks these possibilities, disregards shear-force work, and violates the first energy law since 50 years. The denied but strictly quantified loss of energy (20% for physical $h^{3/2}$; 33.33% at believed $h^2$) violates the first energy law and disregards the force remaining for penetration. Straightforward correction is performed for the dimensions, by replacing unphysical exponent 2. The correction factors $h_{max}^{1/2}$ and 0.8 are applied via joint maximal force to the universal, FE-simulated, (approximately) ISO hardness, and ISO modulus that unduly rely on $h^2$, to give the physically founded values with their correct dimensions. Previous corrected $k$-values obtain $H_{phys}$ directly from the loading curve regression. Previous incomplete corrections are rectified. The new dimensions and daily risk liabilities from ISO versus physics dilemma are discussed, considering the influence on all mechanical parameters from hardness and modulus, regarding technique, biology, medicine, daily life.

**Keywords:** Correction of ISO hardness and modulus; Energy law violations; Failure risks; False materials parameters; False ISO-standards; Indentation exponent; New hardness and modulus definitions; Penetration resistance; Physical consequences; Physical hardness from loading curve

## Introduction

The most basic natural and technical law that can never be dismissed, but must be strictly obeyed, is the energy conservation law. All worlds work on it and must trust in its validity, and that must not be dismissed by any organization. However, ISO and its subsidiary NIST in USA still violate against with ISO standard 14577, claiming the exhaustively complicated mathematical deductions of Sneddon and Love [1,2]. However, these authors obviously missed taking into account the shear-force work, when a rigid indenter is forced to penetrate vertically into a solid. It must be very clear that the pressure (and or plastic deformation) from the rigid indenter against its displaced solid material requires work. Nevertheless, the whole applied force and thus the whole applied energy is still falsely considered to be only acting in vertical direction of the impact. Unfortunately, there was no protest from physics. Rather the work of Oliver and Pharr [3] on the indentation of cones or pyramids was highly acclaimed and adapted by ISO/NIST for ISO 14577. It thus became undisclosed that their assumed relation between force and depth is incorrect and that the hardness and elastic modulus determinations violate the first principle of energy conservation. Such disregard has still been retained till now, even though the unphysical exponent 2 on the depth $h$ had been experimentally demonstrated to be replaced by 3/2 from the present author since 2000 with convincing evidence.

In 1939 and 1965 two mathematicians solved the long standing Boussinesq problem using very complicated mathematics and came (with different constant) to the same exponent 2 on the depth $h$ in relation to the normally applied force in conical indentation when the indenter remains stiff (Figure 1). The Sneddon/Love exponent [1,2] has also been used for partly plastic response (it is a consequence of pressure!) by Oliver and Pharr in 1992 [3], the ISO standard 14577, and finite element (FE) simulations (e.g. ANSYS or ABACUS software), even though the shear force of the conical (similarly effective cone of pyramids) indenter to the environment did apparently not find any concern in physics. Rather numerous fitting procedures were put forward over the years for the excuse, that the exponent 2 on $h$ could not be found experimentally but only with FE-simulations converging to such exponent. Thus, these mathematical deductions (Figure 1) cannot be correct. It did apparently not help that the energetics of the (pseudo) conical indentation was for the first time quantitatively clarified in a publication from 2013 [4] because the experimental exponent on $h$ was consistently found as 3/2 instead of 2 [5]. The thoughtful convincing

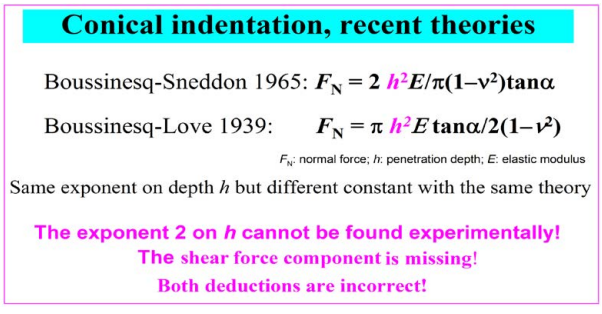


**Figure 1:** Previous highest grade mathematic deductions of load-displacement indentation curves with cones and pyramids.

***Corresponding author:** Kaupp G, University of Oldenburg, Diekweg 15 D-26188 Edewecht, Germany, Tel: 4944868386; Fax: 494486920704; E-mail: gerd.kaupp@uni-oldenburg.de

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physical foundation of exponent 3/2 in Equation (1) that followed pre-published since 2015 [6] requires only first grade mathematics.

## Materials and Methods

The author`s nanoindentations used a fully calibrated Hysitron Inc. TriboScope(R) Nanomechanical Test Instrument with a two-dimensional transducer and leveling device in force control mode after due calibration, including instrument compliance. The samples were glued to magnetically hold plates and leveled at slopes of ±1° 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 [5,7]. 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 whole data set of the loading curve was used for analysis, using Excel(R). 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 with 400-500 or 3000 data points. They were handled with Excel(R). The distinction of experimental and simulated loading curves succeeded by performing the "Kaupp-plot" (1) revealing $F_N \propto h^{3/2}$ (experimental), surface effects and most important phase changes' onset [8]. The necessary force correction to comply with the energy law is made with the physical *k*-value (0.8 times the slope). Only FE-simulated or iterated curves gave linear unphysical $F_N \propto h^2$ plots. The linear regressions were calculated with Excel(R). In the case of phase changes the kink positions were precisely calculated by equating the regression lines before and after the kink. Initial surface effects were, of course, exempt from the linear regressions. Previous penetration resistance values *k* was corrected for complying with the energy/force/depth loss in Figure 2. A 10-figures pocket-calculator was used for the physical calculations, but the final results are reasonably rounded. 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, in order to show their universal obeying to basic mathematics.

## Results

### The mathematical clarification of the energetics upon (pseudo)conical indentation

We proceed analogous to the deduction in Kaupp [4]. In force controlled indentations the total force $F_N$ is linearly applied. This can provisionally be imaged together with an assumed normal parabola (with exponent 2) as is used by ISO etc. in a force versus depth diagram, as obtained by a FE-calculation from the literature (Figure 3). Such normal parabola has the Formula (2). The work of the simulated indentation ($W_{indent}$) gives (3) by integration. The applied work ($W_{applied}$) is the area of the triangle under the applied work (0-$F_{Nmax}$) in (4). Substitution of $F_{Nmax}$ from (2) into (4) gives (5). The ratio ($W_{applied}$/$W_{indent}$)$_{simul}$=0.5/1/3=3:2. That means: only 2/3 (66.67%) of the applied work (and thus also force) would be left for the indent and 1/3 (33.33%) are for the sum of the reversible pressure and the mostly or completely irreversible plastic deformation energies to the environment. Clearly the disregard of 1/3 from $F_N$ when using the false $h^2$ for the calculation of e.g. ISO hardness $H_{ISO}$ and ISO modulus $E_{r\text{-}ISO}$, or universal hardness ($H_{univ}$=$F_N$/$A_{proj}$, where $A_{proj}$ is projected indenter area, also called Martens hardness), is an obvious and severe violation of the basic energy conservation law. The long-known long-range effects and the elastic deformation would require here 1/3 of the applied energy that would be lost for the penetration depth with ISO, FE-simulation, and universal hardness. But a correction for the false exponent is also required.

$$F_{N\text{-}phys}=k\,h^{3/2} \quad (1)$$

$$F_{N\text{-}simul}=\text{const}\,h^2 \quad (2)$$

$$W_{simul\text{-}indent}=1/3\ \text{const}\,h^3 \quad (3)$$

$$W_{applied}=0.5\,F_{N\text{-}max}\,h_{max} \quad (4)$$

$$W_{simul\text{-}applied}=0.5\ \text{const}'\,h_{max}^{\ 3} \quad (5)$$

In order to clarify the unlikely objection that the applied force would be parabolic, we plot here in Figure 4 both applied force and depth side by side against the time as these develop. It is, of course, seen that these develop simultaneously with total $F_N$ linearly but depth

| Material | Indenter | $h_{max}$ | $k$ µN nm$^{-3/2}$ | $r$ | Reference for original data |
|---|---|---|---|---|---|
| PMMA (122 s) | Berkovich | 1.6 µm | 0.0739 | 0.9999 | Lu 2010 |
| PMMA (33 s) | Berkovich | 4.7 µm | 0.2626 | 0.9991 | Cook and Oyen 2007 |
| PMMA (28 min) | Berkovich | 6.5 µm | 0.1554 | 0.9998 | Cook and Oyen 2007 |
| PMMA (33 min) | Berkovich | 6.9 µm | 0.1418 | 0.9999 | Cook and Oyen 2007 |
| *it*-PP (10 s) | Cube Corner | 433 nm | 0.0102 | 0.9997 | Naimi-Jamal and Kaupp 2008 |
| *it*-PP (10 s) | Berkovich | 300 nm | 0.0274 | 0.9997 | Tranchida 2010 |
| Polystyrene (1 s) | stiff Si-lever | 553 nm | 0.9738 | 0.9997 | Kaupp and Naimi-Jamal 2010 |
| Human Bone | Berkovich | 500 nm | 0.3394 | 0.9999 | Weber et al. 2005 |
| Lobster shell | Cube Corner | 270 nm | 0.0259 | 0.9993 | Kaupp and Naimi-Jamal 2010 |
| Pistachio | Cube Corner | 420 nm | 0.0360 | 0.9998 | (Kaupp and |
| Macadamia | Cube Corner | 300 nm | 0.0389 | 0.9995 | Naimi-Jamal 2011) |
| Cherry Stone | Berkovich | 400 nm | 0.0936 | 0.9995 | Kaupp and Naimi-Jamal 2010 |
| Copper (001) | Berkovich | 200 nm | 0.3043 | 0.9999 | Shibutani and Tsuru 2007 |
| γ-TiAl | Cube Corner | 360 nm | 0.2818 | 0.9996 | Zambaldi et al. (2011) |
| InAs | Berkovich | 200 nm | 1.0666 | 0.9999 | Le Bourhis and Patriarche 2005 |
| Aluminum (exp.) | Berkovich | 250 nm | 0.1887 | 0.9994 | Soare et al. 2005 |
| Aluminum | Berkovich | 1.2 µm | 0.2233 | 0.9999 | Naimi-Jamal and Kaupp 2004 |
| ZnO | Berkovich | 200 nm | 1.3100 | 0.9997 | Fang and Kang 2008 |
| MgO | Berkovich | 530 nm | 4.1625 | 0.9999 | Tromas et al. 1999 |
| PDMS | Sphere(192µm) | 3.5µm | 0.001262 | 0.9999 | Ebenstein et al. 2006 |
| PDMS(1:10) | Cone (90°) | 556µm | 0.000878 | 0.9981 | Lim, Chaudhri 2004 |

**Figure 2:** Slightly supplemented table from Reference [5] with corrected penetration resistance *k* (1) (factor 0.8), with unchanged correlation coefficients of various materials, indenters, methods, and authors for the whole length, all without phase transition up to $h_{max}$.

$h$ parabolic. We can thus safely calculate the total applied work from the triangle as in Figure 3 (or Figure 5). Different ways of normal force applications (force controlled, displacement controlled, continuous stiffness, squared progression of the load increments) cannot decrease this applied work. Furthermore, the analysis of strongly creeping loadings (e.g. PMMA data in Figure 2) also gives the unfitted $h^{3/2}$ parabolas (1) with excellent correlation [5] excluding chances to improve the ISO- or FE-indentation efficiency. The formerly forgotten and not considered decreased energy for the indentation and thus also for the actual indentation load part is a striking violation of the first energy law. Only the fraction of the full applied work depends on the exponent on $h$.

But unfortunately we have to respond against continuing strange attacks on the quantitative treatment of conical or pyramidal indentations without any approximations simulations or fittings, despite the publications [4,5]. The probably last denial of the well-established experimental evidence of the exponent 3/2 on $h$ [9] repeats the offence of Troyon (advocating depth dependent broken exponents such as 1.64533 or 1.75285 on $h$ without discussing the incredibly changing dimensions) [10], which is combined with the violation of the first energy law (not considering [4]). Furthermore, Merle [9] tries to invoke the undisputed self-similarity of cones and pyramids as a theoretical argument. But Merle [9] incorrectly claims that this should be in favor of exponent 2. Self-similarity can by no means decide between the exponents in question. The exponent 3/2 is physically founded [6], and all data relying on the false exponent 2 require correction with the dimensional factor $h^{1/2}$. Furthermore, these unduly opposing authors tried to discredit the successful Kaupp-plot ($F_N$ versus $h^{3/2}$) by calling it "Kaupp's double $P$-$h^{3/2}$ fit" [9] ($P$ means force, the same as $F_N$ here), even though the "Kaupp-plot" does not fit at all. They pretend that the kink (phase transformation) in the fused quartz example would have been claimed by intersecting an initial surface effect extrapolation line with the second linear branch, instead of equating the first and second linear branches (more of it in the Discussion). Kaupp has always been identifying surface effects and removing them from the regression.

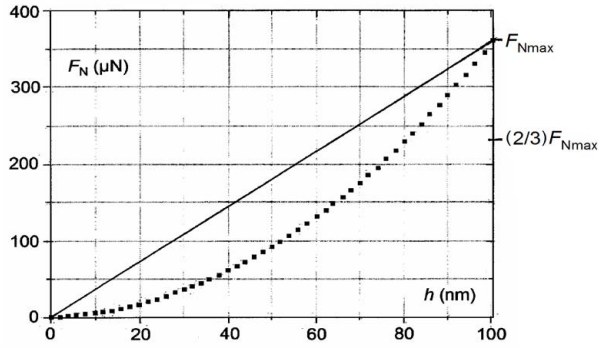


**Figure 3:** FE-simulated force displacement curve for 500 nm thick gold assuming $h^2$ with an ideal Berkovich and our comparison with the linearly applied total work (straight line from zero to $F_{Nmax}$ [4]); the force-corrected parabola would end at the (2/3) $F_{Nmax}$ point; evidently a large part of the applied work would be lost for the indentation; the dotted simulated force curve would precisely follow $h^2$; the simulated data points were taken from Reference [8] (their Figure 3b).

## Experimental and physical basis of pyramidal and conical instrumental indentation

The violation of the basic energy law is connected with the use of unphysical exponent 2 on $h$ with implied assumption that the one third loss of the applied energy ∝ force (Figure 3) would not count for the peak load in the hardness $H$ and modulus $E_r$ calculations that use $F_{Nmax}$ for the start of the unloading curve. The connection is quite simple and direct with the definition of universal hardness for indentations $H_{universal}=F_{Nmax}/A_{proj}$ (where $A_{proj}$ is the projected area of the indenter). This has been worked out in Kaupp [7] with the formula sequence (6) leading to a disproved ISO $F_N \propto h^2$ relation:

$$F_{Nmax}=\pi R^2 H_{universal} \text{ and } R/h=\tan\alpha \text{ gives } F_{Nmax}=\pi h_{max}{}^2 \tan\alpha\, H_{universal} \quad (6)$$

The ISO $F_N \propto h_c{}^2$ relation is also obtained for the ISO-hardness $H_{ISO}=F_{Nmax}/A_{hc}$, where the so called contact height $h_c$ must be adjusted to a standard material in a complicated procedure, including two multiparameter iteration steps [7]. Clearly there are three undisputable flaws against physics with these hardness determinations: 1. the violation of the basic energy law, 2. the use of unphysical exponent and 3. the non-considering of the often occurring phase transformations under load before the chosen peak load is reached, which can only be detected with the Kaupp-plot of (1). The energy law correction will be discussed in the next Section after presenting further support. The dimensional correction will be exemplified in the Sections dealing with the correction of hardness and modulus into physical values.

The convincing physical foundation of exponent 3/2 in the force depth relation (1) [6] (pre-published in 2015) leaves no doubt whatsoever with respect to the present author's analysis of his own and published loading curves from others who wrongly trusted and used the Sneddon/ISO/Oliver-Pharr exponent 2. All details of the loading curves can only be detected when the correct exponent 3/2

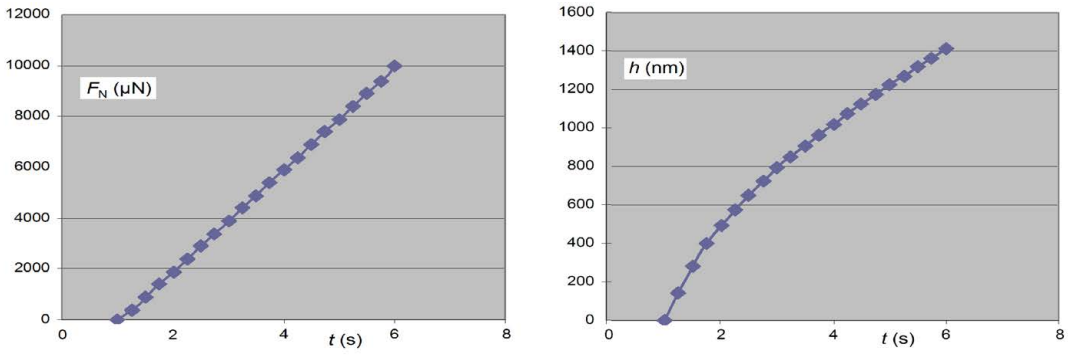


**Figure 4:** Plots of applied normal force $F_N$ and depth $h$ against time in a typical load-controlled NaCl indentation showing linearity of the applied force and non-linearity of depth.

on $h$ is used for the analysis. The details are lost with unphysical plots and more so with data fitting, iterations, or present FE-simulations. Conversely, the physically founded linear $F_N$ versus $h^{3/2}$ Kaupp-plots, as first introduced in lectures since 2000, correct for initial surface effects, reveal phase transformation if they occur within the chosen force range. Furthermore, they detect alternating layers, gradients, pores, defective tips, tilted impressions, and edge interface or too close-by impressions. For example, fused quartz Berkovich indents exhibit the well-known amorphous to amorphous phase transformation [11,12] at about 2.50 or 2.25 mN applied work and 113 or 107 nm depth (analyzed loading curve of Triboscope or CSIRO-UMIS manual, respectively) [11]. This is indicated by a sharp kink in the Kaupp-plot, as it occurs in the chosen loading range [5,11,13].

The force $F_N$ is linearly applied in force controlled experimental indentations. This can again be imaged together with the exponent 3/2 parabola, which is physically founded [6] and experimentally found (Figure 2 [5,11]) and (1). Similar to Equations (2)-(5) deducing $W_{applied}/W_{indent}$ for the wrongly assumed ISO exponent 2 on $h$, the energetic deduction for the physical exponent 3/2 on $h$ is given by the formulas (7)-(9). The physical ratio is thus $W_{applied}/W_{indent}$=5:4. The difference 5−4=1 is for the shear force component exerting pressure and plasticization on the adjacent material. That means: precisely 80% of the applied work and (as $W \propto F$) also applied force $F_N$ is left for the penetration. Thus, 20% is for exerting the sum of pressure and plastic deformation energies to the solid environment. This is considerably less loss for the indentation than if the assumed unphysical exponent 2 would apply (33.33%, see above). The new knowledge is expressively supported with Figure 5 that shows the difference in relation to the Figure 3 for the false exponent.

$$F_N = k\, h^{3/2}$$

$$W_{indent} = 0.4 \text{ const } h^{5/2} \quad (7)$$

$$W_{applied} = 0.5\, F_{Nmax}\, h_{max} \quad (8)$$

$$W_{applied} = 0.5 \text{ const } h_{max}^{5/2} \quad (9)$$

We have now $W_{indent}$=0.8 $W_{applied}$. The basic energy law is thus no longer violated when the applied force $F_N$ (and thus also $k$) is corrected with the factor 0.8. Furthermore the definition of all physical parameters that are related to the indentation force must also not violate the first energy law and require the factor 0.8, provided the exponent correction (2 giving 3/2) has also be performed. Importantly, the now deduced universal 5/4/1 ratio (applied/indent/long-range work) for pyramids and cones is valid for all uniform materials, be they elastic, plastic, migrating, viscous, sinking in, piling up, and flowing. Particular cases are surface effects, gradients, tilted or too tight or edge indentations, pores, micro-voids, cracks, defective tips' effects, and most important kink indicating phase transformation onset. It is valid for all differently angled smooth pyramids or cones with mathematical precision. Any deviations are experimental errors. Surface effects include water layers, gradients, oxides, hydroxides, surface compaction, tip rounding (sometimes compensating other surface effects), and the like. They do not belong to the bulk material and must therefore be eliminated from regressions.

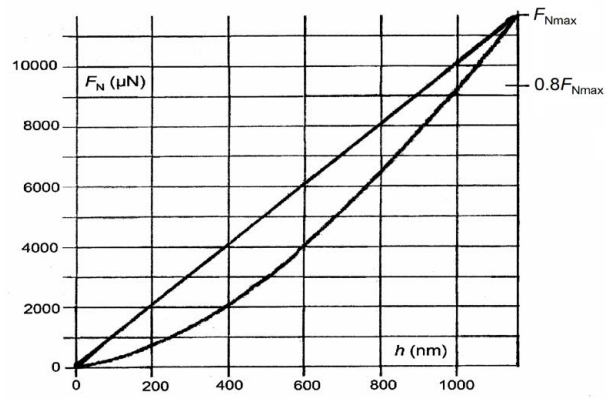


**Figure 5:** Experimental force displacement curve of aluminum (following the physical exponent 3/2 on the depth $h$ [6]) and the comparison with the linearly applied force line, showing the loss of force (and energy) for the indentation depth; the measurement was with a Hysitron Nanoindenter (R); the force-corrected parabola would end at the 0.8 $F_{Nmax}$ point.

## Implementation of the first energy law in instrumented indentation

The energetics of the instrumented depth sensing indentation with pyramids or cones has first been published in 2013 [4] for the $F_N = k\, h^{3/2}$ relation. 20% of the applied work is lost for the indentation with mathematical precision due to the shear-force elastic and plastic work, including sink-in or pile-up. This is universal for all different shapes and materials.

As deduced above, the applied force $F_N$ with the directly proportional otherwise physically correct published parameters (including $H_{phys}$ in [7]) must be corrected with the factor 0.8 (5/4 ratio, 80%) (similarly for $E_{r\text{-}phys}$, see below). Thus, Figure 2 (all with correct exponent 3/2) corrects now the data from the originals in Kaupp [5,7]. Considering the advanced knowledge, this includes all the penetration resistance values $k$ and phase-transformation conversion energies $W_{conv}$ (both correction with the factor 0.8) that were published up to 2016. Not affected are the activation energies and the phase-transformation onsets at characteristic depth, because of cancellation. Also most of the other mechanical parameters from indentations in the literature including ISO-hardness and ISO-modulus are affected. The new knowledge that requires a further specification also for the hardness and modulus definitions requires separate treatment in the next Section below, because these require also the above mentioned dimensional correction.

The tip influence on the $k$-values (Figure 2) and their conversion between different tips has been demonstrated and can be normalized [13]. Creep depends on force and temperature. It is a materials property but does not change the exponent on $h$ of the loading curve, only the penetration resistance $k$. Loading times should thus not exceed 30 s to avoid such influence. Independent creep measurements and corrections must only be performed for most precise rankings of materials. But it is usually much less severe than with the viscoelastic PMMA (strongly diverging from different authors) and certainly for the PDMS values of Figure 2. Indentation times are in fact generally very fast (10-30 s) and creep is mostly slow even at high temperatures, so that a rating along the $k$-values is a good choice already without creep corrections. Creep is mostly not corrected for or published, while thermal drift can be easily corrected for. Creep has however great importance for long-term pressure under heat and for the properties of viscoelastic materials with time dependent behavior. Importantly, the exponent on $h$ remains 3/2 also at indentations of organic crystals with lattice guided anisotropic migrations [13,14].

## Basic energy law and dimensional corrections of indentation

## hardness

A quantitative foundation of conical or pyramidal nanoindentation results as for hardness (and modulus) has to obey the first energy law. All world suffers from such violation that requires correction. The $F_N=k\,h^{3/2}$ relation (1) corrects the fact that only 80% of $F_N$ is used for the indentation with the adjusted $k$-value in accordance with the energy law. The correction of $H_{phys}=k/\pi\ \tan\alpha^2$ (mN/μm$^{3/2}$), as taken from the correct loading curve, where the factor 0.8 is included in the $k$-values, is exhaustive and complies with the first energy law. The physical indentation hardness has unavoidably the dimension (μN/nm$^{3/2}$) or (mN/μm$^{3/2}$) (11). The loading curve provides the easiest, most precise, most rapid and cheap way to obtain the correct physical hardness $H_{phys}$. The deduction of (11) starts with the definition of the universal hardness ($F_N/A_{proj}$) relying on unphysical $h^2$ and violating the energy law. This has to be corrected with the dimensional factor $h^{1/2}$ [that is also required to make Equation (6) concur with physics] for exponent correction to concur with the correct exponent 3/2 on $h$ (1) [7] and the factor 0.8 to concur with the first energy law that is already contained in the penetration resistance $k$. This leads via (10) to (11) after expression of the projected area and insertion of (1) with cancellation of $h^{3/2}$. Importantly, the physical hardness $H_{phys}$ is thus independent of projected area, depth, $F_{Nmax}$, and standard material. It avoids all iterations or fittings or approximations but is experimentally obtained by linear regression and it becomes a genuine physical quantity for the first time. It is also not falsified by undetected phase transformations, because these would show-up in the linear regression. A sharp kink before $F_{Nmax}$ must be absent! The applications of $H_{phys}$ should be very welcome. It is nothing else than a normalized penetration resistance. For example the physical hardness values can be directly obtained from the examples in Figure 2 by using the α-values of the corresponding indenters (Berkovich is ISO-standard).

$$H_{phys}=0.8\,F_{Nmax}h_{max}^{\ 1/2}/\pi\,h_{max}^{\ 2}\tan\alpha^2=k\,h_{max}^{\ 3/2}\,h^{1/2}/\pi h^2\tan\alpha^2 \quad (10)$$

$$H_{phys}=k/\pi\tan\alpha^2\ (\mathrm{mN/\mu m^{3/2}}) \quad (11)$$

The odd appearing dimension mN/μm$^{3/2}$ (also GPa μm$^{1/2}$) of the physical indentation hardness, which does only resemble to a pressure is unavoidable, due to the mathematically fixed shear force component of indentations that cannot be avoided. Nevertheless, indentation remains a very useful particularly precise technique.

Universal hardness, ISO hardness, and FE-simulated hardness would require a factor 2/3 for correction of $F_N$ to give the force for the indentation in order to accept the energy law (Figure 3). But after the necessary multiplication with $h_{max}^{\ 1/2}$ for dimensional correction the force correction becomes 0.8 (Figure 5). However, such corrections of the ISO hardness can only be approximate, because the $h_c$ and thus $A_{hc}$ iterations with respect to a standard material cannot be reverted. Force induced phase-transformations must always be excluded with a Kaupp-plot that at the same time obtains the physical hardness more safely and directly (11).

The equations (12) and (13) show how easy it is to calculate $H_{phys}$ from published $H_{univ}$ or $H_{simul}$ values, provided the $h_{max}$ values for $F_{Nmax}$ are available, and when phase changes are excluded before $F_{Nmax}$ is reached. The corrections are multiplications with $h_{max}^{\ 1/2}$ for the correct exponent 3/2 and factor 0.8 for the force loss.

$$H_{univ}=F_{Nmax}/\pi\ \tan\alpha^2\ h_{max}^{\ 2} \quad (12)$$

$$H_{univ\text{-}corr}=H_{phys}=0.8\,h_{max}^{\ 1/2}\,F_{Nmax}/\pi\ \tan\alpha^2\ h_{max}^{\ 2} \quad (13)$$

This is exemplified in Table 1 with a numerical example from a published indentation onto aluminum, where $H_{ISO}$ and both the FE-simulated $H_{simul}$ (ANSYS software) with exponent 2 on h and the experimental Berkovich loading curves are published (falsely claimed exponent 2 but according to the Kaupp-plot determined with exponent 3/2 on $h$) [15]. Any universal hardness ($H_{univ}$) treatment would be the same as the one for $H_{simul}$. The published loading curve was also provisionally analyzed as $F_N$-$h^2$ plot but only used for numerical achievement of the conversions.

Entry 1 in Table 1 gives the $H_{phys}$ from the analyzed loading curve (11), which is certainly the most reliable value. It does not rely on $F_{Nmax}$, $h_{max}$, any $h_c$ or $A_{hc}$ and it secures the absence of a phase change up to the maximal force. And it compares with $H_{ISO}$ and the hardness values that derive from $H_{simul}$ with various stages of correction.

Entry 2 shows that $H_{ISO}$ exhibits a far too high value and an unphysical dimension. The energy correction for leaving exponent 2, removing only the energy law violation, decreases the value insufficiently, still with the unphysical dimension mN/μm$^2$. A value for $h_{max}$ is not available for a final correction. When exceptionally a guess were tried that it might be in a 0.25 μm region one would guess a further decrease that would look like 0.239 with the changed dimension mN/μm$^{3/2}$. This would be in the region of $H_{phys}$ although with all reservation, because it is only a free guess only indicating the direction. This show the difficulties for the conversion when $h_{max}$ for the used $F_{Nmax}$ is not reported. It is thus much easier to apply the Kaupp-plot to the loading curve (1). We renounce of including the uncorrected simulated value (0.6016 mN/μm$^2$).

Entry 3 gives only the exponent correction of FE $H_{simul}$ (ANSYS-software) that was probably obtained by using Young's modulus E (either known or iterated) input, with converging criterion to exponent 2 on h.

Entry 4 gives only the energy correction with a rather high value. Table 1 show that neither the exponent correction for exponent 2 alone nor the energy correction (Figure 3) alone (removing energy law violation) is sufficient.

Entry 5, finally with both exponent correction and then smaller energy correction factor for h$^{3/2}$ (Figure 5) provides $H_{simul\text{-}phys}$, with

| Number | Technique | $h_{max}^{\ n}$ | $k$ or $h_{max}$ [(a)] | Hardness calculations and corrections |
|---|---|---|---|---|
| 1 | Experimental linear regression | $h_{max}^{\ 3/2}$ | $k$=5.9540(mN/μm$^{3/2}$) (energy corrected)[(b)] | $H_{phys}=k/\pi\tan\alpha^2$=0.24295(mN/μm$^{3/2}$) independent on $F_N$ and $h_{max}$ (no phase trans.) |
| 2 | Experimental with 2/3 factor | $h_{max}^{\ 2}$ | - - | $H_{ISO}$=0.716 (GPa) x (2/3) ≈ 0.477 (mN/μm$^2$) (unphysical dimension) $h_{max}$ not known |
| 3 | FE-simul. $h_{max}^{\ 1/2}$ no energy corr. | $h_{max}^{\ 2}$ | $h_{max}$=251.984 nm | $H_{simul\text{-}corr1}$ (as $H_{univ}$)=$F_{Nmax}/\pi\tan\alpha^2 h_{max}^{\ 3/2}$=0.2977 (mN/μm$^{3/2}$) (energy law violation!) |
| 4 | FE-simul. 2/3; no exponent corr. | $h_{max}^{\ 2}$ | $h_{max}$=251.984 nm | $H_{simul\text{-}corr2}=2F_{Nmax}/3\pi\tan\alpha^2\,h_{max}^{\ 3/2}$=0.4011 (mN/μm$^2$) (wrong exponent) |
| 5 | FE-simul, 0.8, and $h_{max}^{\ 1/2}$ | $h_{max}^{\ 2}$ | $h_{max}$=251.984 nm | $H_{simul\text{-}phys}$=0.8 $F_{Nmax}/\pi\tan\alpha^2\,h_{max}^{\ 3/2}$=0.2382 (mN/μm$^{3/2}$) |

**Note:** [(a)]Simulated parameters are not italicized; [(b)]correction factor 0.8.

**Table 1:** Comparison and correction of unloading $H_{ISO}$ and FE-simulated $H_{simul}$ loading curves of Al on Si [15] with the physical $H_{phys}$, which is in accordance with the energy law.

surprisingly good match (2%) with $H_{phys}$. The surprisingly close coincidence of $H_{phys}$ and $H_{simul\text{-}phys}$ supports the numerical correctness of the non-fitting (!) straightforward deduction and it also reminds the unbeatable precision of the Kaupp-plot's linear regression (Figure 2). The close correspondence with $H_{simul\text{-}phys}$ in this case should however be tested for generality, because this single example could be fortuitous when considering the parameterizations and iteration procedures at FE simulations.

Importantly, the striking dilemma of ISO with physics persists with the false dimension of too large $H_{ISO}$ and unphysical dimension. All of the values and dimensions of the mechanical parameters that depend on it are severely wrong, also those that depend on wrong ISO elastic modulus $E_r$ (see next Section). Clearly, Table 1 and Equations (12) with (13) show an easy way for straightforward corrections of $H_{univers}$, probably $H_{simul}$, and with reservation $H_{ISO}$, provided the $h_{max}$ values are known. However, despite the straightforward corrections none of them can handle the very often occurring and so important phase transformations under load (here they were experimentally excluded with Kaupp-plot).

## Basic energy law and corrections of indentation elastic modulus

Also the correction of unphysical $E_{r\text{-}ISO}$ into a physical value is essential, because elasticity is a technically important materials property. Young's moduli $E$ are required for the deduction of numerous mechanical qualities and for example increasingly as input parameter for numerical FE-simulations, often including FE-iterations with E-Y pairs as free parameters (where Y is yield strength). The determination of the elastic modulus requires the unloading stiffness $S=dF_{Nmax}/dh_{max}$ from the pressure to the displaced material that must be separated from the plastic response. This is achieved for $F_{Nmax}$, which is a joint quantity of loading and unloading curves. Thus, there must again be corrections for dimension adjustment with $h^{1/2}$ [for not violating (1)] and for shear force loss during the loading (for not violating the energy law). These are not applied in ISO 14577 that applies the Oliver-Pharr iterations [3]. Thus, the slope correction for $E_{r\text{-}ISO}$ (14) requires again the exponent correction with $h^{1/2}$ and then the force correction factor 0.8 (Figure 5) to comply with the energy law for obtaining $E_{r\text{-}phys}$. This gives via (14) $E_{r\text{-}phys}$ (15) in complete correspondence with the necessary treatment of $H_{ISO}$ (this replaces the incomplete formula 11 in [7]). Again one must be certain that the unloading was performed at $F_{max}$ before any onset of a force derived phase transformation had occurred. By comparing (14) and (15) the correction factors are found to be 0.8 and $h_{max}^{1/2}$. The corrections for obtaining physical modulus values with changed dimension is simply by multiplication with 0.8 $h_{max}^{1/2}$. The unloading stiffness $S$ and $h_{max}$ (before creep) must be known. This is another dilemma between ISO and physics.

$$E_{r\text{-}ISO}=S\,\pi^{1/2}/2\,A_{proj}^{1/2}=(dF_N/dh)_{max}/2\,h_{max}\tan\alpha \quad (14)$$

$$E_{r\text{-}phys}=0.8\,\pi^{1/2}\,h_{max}^{1/2}\,S/2\,\pi^{1/2}\,h_{max}\tan\alpha=0.8\,S/2\,h_{max}^{1/2}\tan\alpha\ (mN/\mu m^{3/2}) \quad (15)$$

## Discussion

The extremely complicated mathematical deductions of Sneddon/Love ([1,2]; Figure 1) for the conical or pyramidal indentations did not consider the energetics of the process, as illustrated with the Figures 3 and 5. And there was no protest from physicists. Almost all involved people followed Sneddon [1], Oliver Pharr [3], and ISO 14577 all with violating the first energy law for more than half a century. The general acceptance for half a century of the implied claim that pressure formation and plasticization could be workless achieved is hard to understand. It is apparently the result of hype upon the publication [1] that unfortunately was believed by ISO/NIST. The simple equations as derived starting in 2000 ([14] and before in lectures and in refused manuscripts) and the point by point unraveling of the field until now against strong impediments did not help. The newcomers had to obey ISO 14577 and many very complex rules, and they used the software of the instrument suppliers that had to trust in the ISO/NIST-standards. By doing so they forgot to think on the physical foundations. Thus, the basic formulas (3)-(5) and (7)-(9) that essentially rely on the experimentally (since 2000) (Figure 2) and physically founded (since 2015) Equation (1) [6] found much refusal, various excuses for not experimentally finding exponent 2 with fittings, multi-parameter iterations, and simulations. The actions against the elementary algebraic treatment without any fitting/iterating/simulating were undue repetitive offenses. Rather acknowledgement had to be expected because everything became much easier and quantitative on a sound physical basis with simple closed mathematical formulas, proving the universal validity.

Apparently, nobody else (not even textbook or tutorial writers) asked themselves why all of the applied normal force with cones or pyramids is claimed to be used for the indentation depth, even though the loading curve proceeds not linearly but parabolic. The obvious answer is that well-known long range effects and pressure formation to the environmental solid material require energy that is lost for the indentation depth. When this energy/force/loss was quantified and finally (after difficulties with anonymous Reviewers) published in 2013 [4] with the universal loss of 1/5 for the physical (1) and 1/3 for unphysical (2) equations, there was discussion about the validity for comparing applied work and indentation work. But these proceed at the same time to the same endpoint $F_{max}$. Surprised about the ease of the mathematical deduction and the strict and universal result, requiring difficult necessary changes, there were objections and much open discussion in plenary lectures from the audience with the guess that all of the linearly applied force might instead go along the parabolic curve during the experiment. This prevented the opponents from recognizing that the first energy law was evidently violated. The linearity of the applied force is however also evident, simply from the additional applied force $F_N$ versus time plot in Figure 4.

The undue opposition against straight forward physics and algebra is surprising even after it was very clear with Kaupp [4] that the ISO-system violates the first energy law (the present author could not dare to verbally express the energy law violation at that time). The offenses have been continuing. For example, the opposing manuscript [9] was received at Scanning on May 27, 2014, whereas the clarifying manuscript [4] was received at Scanning on October 4, 2012 and published on February 25, 2013. The content of Kaupp [4] had thus to be taken up again in Kaupp [7] with more details, because the authors, reviewers, and editors of Merle [9] continued violating the basic energy law. And the Merle [9] continued arguing against the most precise Kaupp-plot that actually was the basis for the quantification of the violation. The opponents tried with iterated own loading curves of fused quartz. But when doing it correctly, even the invoked curve in Troyon [10] would roughly reproduce the well-known transformation onset, despite its using a blunt tip that gave an unusually long initial effect. And Merle [9] tries again with a false intersection at its microindentation "Kaupp-plot" (up to 300 mN and 1600 nm) where the region with all of the nanoindentation details is almost totally obscured in a short unstructured part of it. The false intersection with

a remote line far away from the plot is useless. But it is used for falsely criticizing the Kaupp-plots that never used or use such faulty tricks. When properly looking at this linear plot in Reference [9] with a ruler, one recognizes an intersection of two straight lines at about 175 mN and 1225 nm, which the authors do neither trace nor recognize. Four possibilities exist for this kink very close to the plot: either a new high-load phase transition of fused quartz occurred, or a smoothness defect of the tip was present at this depth, or a remote crack at such deep impression was formed, or the impression was too close to an edge/interface/impression. Furthermore, these authors claim and draw a straight single line for their unphysical so called "$P$-$h^2$ fit with 0.999 fit quality". However, despite their claimed "three-nines fit", their depicted unphysical "P-$h^2$ fit" gives two roughly linear branches, intersecting in the region of 60-70 mN force (that is far away from surface effects). This deviation from the claim is easily "overlooked" without a ruler in a wide pencil stroke representation at totally false depth-square scaling (better seen when more precisely drawn, the first part steeper and cutting at small angle). This shall only be a necessary contradiction to the false claim of linearity for a "P-$h^2$ fit" trying to discredit our simple algebraic treatment on a sound physical foundation. Fitted or FE curves, converging with $h^2$, must not be used for denying thoughtful and repeatable physically founded [6] and experimental Equation (1). Only untreated experimental loading curves are able to detect surface effects, the important phase changes, conversion energies, etc., when using the physically founded exponent on $h$.

A problem might arise when fitted, iterated or FE-simulated curves and experimental loading curves might be mixed up in publications. However, when experimental force data are plotted with or fitted to the non-physical $h^2$, the deviations from a straight line might appear minor for example as in Merle [9]. Also a minor endothermic phase change slightly levels the unphysical $F_N$-$h^2$ trial-plot with respect to the stronger curved appearance without phase change [5,11]. Such leveling behavior of the test material fused quartz might have strengthened the belief in $h^2$, but it reflects the inability to find phase transformation with the physically wrong exponent 2 on $h$. All of the important details of nanoindentation are lost with $h^2$. But the kink at $F_N \approx 2.4$ mN (Berkovich) and initial surface effects of the fused quartz standard are easily seen by sharp kinks with the precise Kaupp-plot (1) in nanoindentations, notwithstanding the cases of later or further phase changes in microindentations (e.g. NaCl in [5,11]). But there is no excuse for using the unphysical exponent and thus denial of the phase transitions if these occur, combined with the violation of the first energy law.

The readers of Kaupp [4] and the attendants of the present author's lectures on numerous worldwide conferences were repeatedly urged to think about the unexpected and surprisingly easy deduced energetic facts (2)-(6) and (7)-(9) but the expected response of the scientific establishment is still missing. It appeared unlikely that all of the scientific Celebrities and their successors including textbook authors, ISO/NIST, and numerous anonymous referees have, consciously or not, been violating the first energy law for more than 50 years. Hesitation to use only the normal force left for the indentation depth was thus advisable, before any non-apparent compensation effect for saving the energy law was excluded in the desperate situation. Publications of the truth should stay as close as possible with the current indentation theory unless all objections are removed. Clearly, the believers in exponent 2 on $h$ could for themselves have easily performed the deductions as in Equations (1)-(5) and could have tried to change their minds because of this inexcusable energy law violation. But they did not try to take into account the always occurring energy loss. Based on their believed exponent 2 on $h$ it would have amounted to 1/3 (33.33%) of $F_N$ due to the work and force proportionality, as shown above with the trial Equations (2)-(5). And they would have found that the violation is also programmed and used in FE-simulations. They refused till now to accept the undeniable wealth of the Kaupp-plot and the physical deduction for the correct exponent 3/2 on $h$ [6] that finally proves energy/force loss of 1/5 (20%) according to Equations (7)-(9), as only the physical exponent is correct. Since ISO/NIST have been reluctant to change their minds, or to announce reconsideration with an alert, there was the urgent preliminary publication in Kaupp [7] for expressively naming the incredible claim of workless pressure formation and plasticization as "violation of the basic first energy law". This is now completed with valid transformation formulas for obtaining the physical values and the necessary conditions for that from unphysical publications. Furthermore, the most easy and precise $H_{phys}$ determination by linear regression of the loading curve (1) (hitherto strongly refuted Kaupp-plot) with energy-based correction is now again strongly advocated for.

## Conclusions

The still not settled dilemma between ISO and physics with respect to ISO 14577 (not even an alert has been filed yet) is unbearable due to its enormous risks for science and daily life's safety. It appears unbelievable and even desperate that the first energy law was drastically violated for more than 50 years and none of the physicists protested against such habit. Everything is easily deduced with first grade algebra, avoiding fittings, iterations, simulations, and approximations, making everything much more easy. Hardness is now obtainable by linear regression, no longer by iterations, fittings, approximations, and simulations that are not ready for a controlling assessment. The physical indentation hardness $H_{phys}$ (mN/μm$^{3/2}$) is now for the first time a genuine physical quantity, obeying Equation (1) and the first energy law. The same is true for the indentation modulus $E_{r\text{-}phys}$ (mN/μm$^{3/2}$). The complete, more precise deduction than in Kaupp [7] reveals also the simple conversion from $E_{r\text{-}ISO}$. Only the quantitative indentation on the physical basis reveals numerous otherwise impossible applications. Examples are phase change [4,5,16], conversion energies [4,16] and activation energies [16] of materials, all on the basis of the so-called Kaupp-plot (1) that also checks for correctly performed indentations and provides extrapolation facility up to recognized phase change qualities under pressure. Furthermore, it reveals a large number of special materials' properties and indentation errors that are named above. But it is still being heavily suppressed by the ignoring establishment, including ISO and some anonymous Reviewers with incredible unqualified wording instead of acknowledging this wealth.

The liability with unphysical calculated materials' properties is totally unclear at the present dilemma between ISO and physics, because all safety engineers are falsely trained. That means, the issue counts for every days safety unless ISO files at least an urgent alert. Everybody knows how many materials fail shortly after the warranty period, certainly not purposeful but often with falsely calculated materials. Even worse, falsely calculated components like poorly adjusted medicinal implants or larger scale composites can produce disasters. There is good reason why passenger traffic airplanes require frequent safety checks and complete replacement of all parts within 2 years. For example $h$ goes with $F_N^{2/3}$ not with $F_N^{1/2}$ [5] with all implications for fatigue, and wear, to name a few.

Despite the highly comprehensive results of this paper and numerous worldwide lectures on conferences the ISO versus physics dilemma still remains. The physical indentation $H_{phys}$ and $E_{r\text{-}phys}$ dimensions that only

resemble pressures is perhaps difficult to understand at first glance. But it is real and the reasons have been discussed. Importantly this does not detract from indentation as a very precise and reproducible technique, when properly executed, checked, and algebraic evaluated, that means without fittings, iterations, and simulations.. Rather the unavoidable dimensional changes have an enormous bearing for science and practice. The not fitted and not iterated physical quantities must be used to redefine the numerous further mechanical parameters that were deduced from unphysical $H_{ISO}$ or $E_{r\text{-}ISO}$. Further studies are necessary and further important insights are to be expected when the violation of the first and most basic energy law will be removed also for the deduced parameters. This should help for a better understanding and open new horizons. Also textbooks must be rewritten for the sake of physics, compatible materials sciences, and new insights. Since there was the violation of the first energy law, the new results will prove to be more compatible with all related techniques that do not violate physical laws, which is very desirable. The quantitative indentation at the now physical basis has the indispensable advantages of being precise, and in accord with basic principles. This is promising and cannot be denied. The further advancement on the physical basis is a very urgent task that must be pursued, hopefully soon also with ISO/NIST against all of the incredible resistance, because violating the first energy law is an inexcusable fault.

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