

## Challenge of Industrial High-load One-point Hardness and of Depth Sensing Modulus

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### Abstract

The physics of industrial single-point force indentation hardness measurements (Vickers, Knoop, Brinell, Rockwell, Shore, Leeb, and others) is compared with the depth-sensing nano, micro, and macro instrumental hardness technique that provides several further mechanical parameters, when using the correct force/depth curves exponent 3/2 on the depth of the loading curves. Only the latter reveal phase change onset with transition energy, and temperature-dependent activation energy, which provides important information for applications of all types of solids, but is not considered in the ISO or ASTM standards. Furthermore, the high-load one-point techniques leave the inevitably even stronger and more diverse consecutive phase-transformations undetected, so that the properties of pristine materials are not obtained. But materials are mostly not (continuously) applied under so high load, which must lead to severe misinterpretations. The dilemma of ISO or ASTM standards violating the basic energy law, the dimensional law, and denying the occurrence of phase changes under load is demonstrated with the physics of depth-sensing indentations. Transformation of iterated ISO-hardness and finite element simulated hardness to physical hardness is exemplified. The one-point techniques remain important for industry, but they must be complemented by physical hardness with detection of the phase transformation onset sequences for the reliability of their results.

The elastic modulus  $E_{ISO}$  from unloading curves as hitherto unduly called "Young's" modulus has nothing in common with unidirectional Young's modulus according to Hook's law, because the skew tip faces collect contributions from all crystal faces including shear moduli, while iteration fit is to Young's modulus of a standard. Unphysical and also physically corrected multidirectional indentation moduli mixtures of mostly anisotropic materials and there from deduced mechanical parameters have no physical basis and none of these should be used any more. A possible solution of this dilemma might be the use of indentation- $E_{phys}$  and bulk moduli  $K$  from hydrostatic compression measurements. The reasons for obeying physical laws in the mechanics of materials are stressed.

**Keywords:** Brinell hardness; Elastic modulus; Force-depth curves; Hook's law; ISO and ASTM standards; Macroindentation; Physical hardness and modulus; Rockwell hardness; Ultrasound; Vickers hardness, Young's modulus

### Introduction

While present depth-sensing indentation hardness and modulus determination is obtained by nano- and sometimes micro-indentation (nN to  $\mu$ N and mN), instrumented macro-indentation is also possible up to 80N. Industrial non-depth-sensing techniques still concentrate to longer known Vickers (HV), Knoop (HK), Brinell (HB), Rockwell (HR), Shore, rebound LH (Leeb), or more specialized macro-hardness tests. These measure the impression diagonal, or diameters, or final depths under specified conditions although with subsets for certain types of materials. One subset of HV is the UCI technique (ultrasonic contact impedance, requiring elastic modulus  $E_{eff}$ ) measuring vibration damping of a swinging stick with a Vickers diamond at the end, as inserted at a predefined load. All these single-point high-force techniques require 1:1-calibration with test plates of closely related materials of "known" hardness, also for canceling out not specified tip end radii. Several hand-held devices exist, which is practical in the steel industry. Hand-held equipment includes UCI, Leeb, Rockwell clamp, Brinell clamp, Brinell Poldi hammer, etc. All of these techniques use rather empirical definition and ISO (International Standard Organization) or the now compelling ASTM (American Society for Testing and Materials) standards. Between HV, HK (low load range), HB, HRB, and HRC exist approximate conversion equations. This indicates relationships between them. The equipment software usually calculates and displays most of the different types of macro hardness. However, conversion between them is often not precise enough for construction purposes, notwithstanding the sometimes large experimental uncertainties, due

to often low reproducibility between different user sites. And there is serious non-compliance with basic physics.

The comparison with depth-sensing instrumented indentation according to ISO 14577 where three major flaws occur in the universal, ISO, and finite element (FE) simulated hardness, is difficult. The instrumented depth-sensing could recently be corrected for providing the physical hardness, eqn. (1) (where  $k$  is the slope of the so called "Kaupp-plot" eqn. (2)) by removing three physical flaws inherent to ISO 14577 [1-3].

$$H_{phys} = 0.8k/\pi(\tan\alpha)^2 \quad (1)$$

$$F_N = kh^{3/2} \quad (2)$$

Corresponding violations of physical laws have not yet been considered in the single-point-load techniques, but these must equally exist. This bears an important risk for the mechanics quality of industrial goods. A prevailing source of uncertainty is the non-considered phase transformation of materials that change the material's hardness and

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other mechanical properties, under the very large local pressure. It is well-known [1,3] that phase changes occur already at nano-indentation and lower micro-indentation. They must therefore be even more common in the macro range. Furthermore, the possibilities for detection of hidden horizontal cracks (except when these occur upon unloading) are not evident. All of the industrial indentation techniques also penetrate vertically onto flat surfaces, but now with a defined holding time at the predetermined force. Creep is assumed to be negligible. Some of these macro-techniques (HV, HK, HB) measure diagonals or diameters of the impressions that is left at the surface, others (HR, Shore) the indentation depth. But final depths can also be calculated from the indenter geometries in the former cases. The physical flaws as detected in the instrumented depth-sensing should be the same in all macro-hardness tests. Clearly, the depth relates to the diagonal or diameter left at the surface. So there is no principal difference to more precise depth sensing, except that the applied forces are usually very much higher. Very detailed and constantly refined ISO and ASTM standards are available. A comparison between these and the depth-sensing techniques is thus in urgent order, by applying the physical news from the nano- and micro-indentations [3].

Similar difficulties with elastic moduli concern only the depth sensing unloading. The same dimensional energetic and phase change violations of ISO standards can be principally corrected. However, it turns out, there is not the claimed correspondence of ISO or physical indentation moduli with Hook's Young's moduli, so that  $E_{ISO}$  should no longer be iterated (Oliver-Pharr method), falsely called "Young's" modulus, and used. Even  $E_{phys}$  is only a counterpart of  $H_{phys}$ , the physical hardness. It will however be suggested to use bulk moduli instead.

## Materials and Methods

The nanoindentations onto a polished optical disc 2 mm thick NaCl single crystal (purchased from Alpha Aesar GmbH Co KG, Karlsruhe, Germany) were performed at a Triboindenter<sup>(R)</sup> with AFM of Hysitron Inc, Minneapolis, USA, with proper calibration at 23, 100, 300, and 400°C (average of eight measurements). The author's nanoindentations used a fully calibrated Hysitron Inc. Triboscope<sup>(R)</sup> instrument with AFM in force controlled mode also with a Berkovich diamond ( $R=110$  nm). The cited literature data have been carefully searched and interpreted in view of the generally deduced physical laws, in accordance with validated experimental data. Phase changes under load are detected by kink-type discontinuity [4] in so-called "Kaupp-plots" according to eqn. (2) [1-5]. The precise intersection point is obtained by equating the regression lines before and after the onset of the phase change. The regression coefficients are calculated with all 400-500 or 3000 original data point pairs using Excel<sup>(R)</sup>, but

excluding those from initial surface effects. Digitizing 50-70 almost uniformly arranged data pairs were obtained from published loading curves with the aid of the Plot Digitizer 2.5.1 program (www.Softpedia.com). The distinction of experimental from FE-simulated loading curves succeeded with the "Kaupp-plot". The necessary correction to comply with the energy law [1,3] is by multiplying the slope  $k$  ( $mN/\mu m^{3/2}$ ) with 0.8.

## Results

### Dilemma between ISO standards and physics

The comparison of depth-sensing instrumented ISO-hardness with non-depth-sensing single point high-load techniques reveals undeniable physical similarities. The industrially used macro indentation techniques are governed by the same physical laws as depth-sensing nano to macro indentations. Unfortunately, present ISO standards are at variance with the corresponding physical laws [1-3] and the possible corrections of previously published indentation data require a detailed discussion here. The physical requirements for single-point load indentations reveal equally from the precisely determined facts of the better controlled depth-sensing continuous indentations, including the macro-indentation ones.

Table 1 compares the depth-sensing hardness values of  $H_{phys}$ ,  $H_{ISO}$ , and  $H_{simulated}$  to demonstrate the importance of correct depth-sensing evaluation. It is also shown how the latter two can be corrected, provided that the loading curves were published as for example in ref. [6]. This is a practical application for the conversions of FE-simulated or ISO hardness values (energy law violations and incorrect exponents) into physical hardness.

Entry 1 shows the correct value  $H_{phys}$ , according to the Kaupp-plot with linear regression from the experimental loading curve in ref. [6].

Entry 2 deals with the published iterated  $H_{ISO}$  value that enormously differs in value and dimension. The difference is still very large when the energetic law violation is removed (based on the falsely believed " $h^2$ " the energy or force loss for the indentation calculates to 33.33% energy law violation) (cf. refs. [1,2]). This is incomplete correction. It is not clear, which  $F_N/h$  pair was used in the  $H_{ISO}$  iteration. Complete correction would also suffer from the exhaustive iterations that cannot be reverted.

Entry 3 deals with the FE-simulated hardness without the necessary corrections: again, a large deviation in value and dimension from  $H_{phys}$ .

Entry 4 demonstrates only the dimensional correction, as  $h_{max}^2$  was used instead of  $h_{max}^{3/2}$  for the relation with  $F_{max}$ , but it is clearly not

| Entry | Technique                                      | $h_{max}^n$     | $k$ or $h_{max} F_{max}^{(a)}$                               | Hardness calculations and corrections   |
|-------|--|-----------------|--|---|
| 1     | Experimental curve linear regression           | $h_{max}^{3/2}$ | $k=5.9540(mN/\mu m^{3/2})$ (energy corrected) <sup>(b)</sup> | $H_{phys}=k/\pi \tan \alpha^2=0.24295(mN/\mu m^{3/2})$ Independent on $F_N$ and $h_{max}$ (no phase trans.)                   |
| 2     | Iterated $H_{ISO}$ with 2/3 factor             | $h_{max}^2$     | --   | $H_{ISO}=0.716(GPa) \times (2/3)=0.477(mN/\mu m^2)$ (still unphysical dimension, $h_{max}$ unknown)                           |
| 3     | FE-simulated not corrected                     | $h_{max}^2$     | $h_{max}=0.250 \mu m$<br>$F_{max}=0.912 mN$                  | $H_{simul}$ (as $H_{univ}$ )= $0.6016(mN/\mu m^2)$  |
| 4     | FE-simul. $h_{max}^{1/2}$ no energetic corr.   | $h_{max}^2$     | $h_{max}=0.250 \mu m$<br>$F_{max}=0.912 mN$                  | $H_{simul}$ (as $H_{univ}$ )= $F_{Nmax}/\pi \tan \alpha^2 h_{max}^{3/2}=0.2977(mN/\mu m^{3/2})$ (still energy law violation!) |
| 5     | FE-simul. 2/3; no exponent corr.               | $h_{max}^2$     | $h_{max}=0.250 \mu m$  | $H_{simul-corr2}=2 \times 0.6016/3=0.4011(mN/\mu m^2)$ (wrong exponent)   |
| 6     | FE-simul., $h_{max}^{1/2}$ and energetic corr. | $h_{max}^2$     | $h_{max}=0.250 \mu m$<br>$F_{max}=0.912 mN$                  | $H_{simul-phys}=0.8 \times 0.2977=0.2382(mN/\mu m^{3/2})$   |

<sup>(a)</sup>simulated parameters are not italicized; <sup>(b)</sup>energy correction factor 0.8.

**Table 1:** Comparison and correction of  $H_{phys}$ ,  $H_{ISO}$  and FE-simulated  $H_{simul}$  loading curves of Al [6] including the corrections in accordance with the exponential and energy laws; extended table from ref. [3].

sufficient.

Entry 5 similarly reveals that only the removal of the energy law violation (for believed  $h^2$  only 2/3 of  $F_N$  is available) is not sufficient.

Entry 6 shows that only both corrections ( $h^{1/2}$  and thereafter 0.8) give a good value. But it has to be checked if all FE-simulations with different iterations will give equally good correspondence with the correct experimental  $H_{phys}$  value. All of the corrections in Table 1 are equally valid for the one-point non-depth-sensing macro techniques. However, there is another very important flaw: phase changes under load. Their occurrence and onset load can only be detected by depth-sensing indentation and Kaupp-plot.

As hardness is for the first time a physical quantity, there is no possibility to change the physical hardness dimension say by division with  $h^{1/2}$ , which would mean to accept the dimensional violation. The inherent dimension has its meaning for all applications of the  $k$ -value (force/depth<sup>3/2</sup>, mN/ $\mu\text{m}^{3/2}$ ) that cannot be dismissed. These include depth-force relation (mar, wear, tribology), physical deduction with elementary mathematic, adhesion work, pull-off curves, safe ratings of materials, correlation coefficients with >3 nines or less noisy with >4 nines, quantitative far-reaching energy, phase transition onset, indentation work, compatibilities, transformation energies, activation energies, creep, size effects, maximal load for reasonable unloading curves, initial surface effects, high sensitivity by linear regression, tip normalization, tip rounding effects, materials gradients, inhomogeneous materials, geodes, crystal defects, edge interface, too close impressions, grain boundaries, cracks, alternating, improvement of FE-simulations, avoidance of polynomial iterations or varying broken exponents, correct visco-elastic-plastic parameters, nanopores, micro-voids, alternating layers, blunted tip effects, correction of false mechanical parameters that rely on  $h^2$ , elementary mathematics instead of iterative fitting, avoiding violation of the basic energy law with factor 0.8, failure risks with false mechanical parameters, tilted impressions, faulty standards with phase change, all types of solid materials and

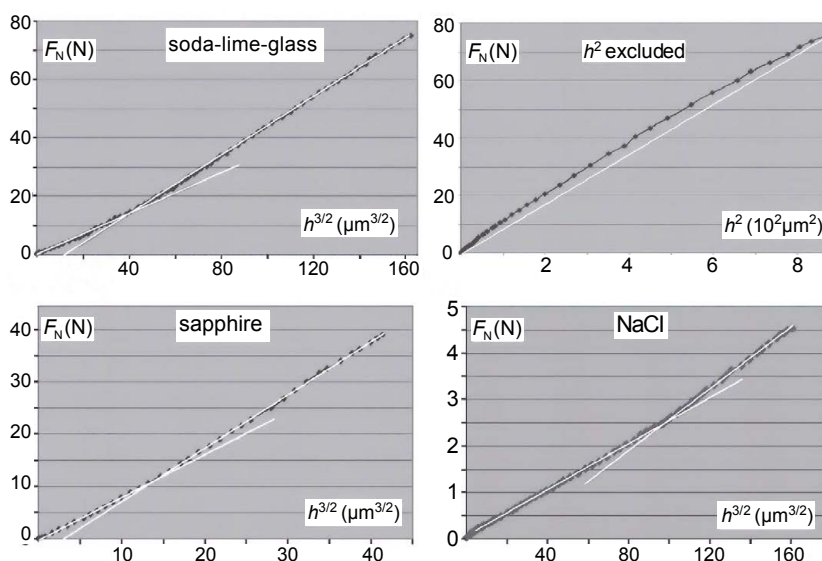
plasticization types [1-5,8], quantitative sound mathematical basis, universal validity, distinction of FE-simulated from experimental loading curves, no denial of phase changes, and daily risk with unphysical mechanic parameters [3].

### Macroscopic depth-sensing, hardness and phase transitions

Depth-sensing nanoindentation extends up to 10 mN load, micro-indentations up to 1 N, mostly with Berkovich indenter. Extensions to macro-indentations have been achieved with Vickers up to 80 N for soda-lime glass [7], but rarely repeated. Eqn. (2) has been experimentally secured and physically deduced. It secures Kaupp plots for all of the force ranges [2,5]. The dilemma of physics and ISO 14577, still believing in " $h^2$ ", is clearly evident from Figure 1. It indicates exclusion of  $h^2$  and phase transitions of soda-lime-glass, sapphire, and sodium chloride in the macro-indentation range (the nano-ranges are "hidden" at these macro-indentation ranges). The kink-type discontinuities (the phase transition onsets) are at 15.37, 10.43, and 2.469 N load, respectively.

The most important advantage of macro-depth-sensing is the detection of secondary phase transitions at very large forces. In the case of NaCl, the also endothermic fcc to bcc transition at 4.233 mN [8] is hidden at that scale. The same is true for a transition onset of sapphire at 26.5 mN load and for soda-lime-glass at 4.81 mN [5]. The other macro-indentation techniques have the disadvantage that they cannot detect phase transition onsets: they practically always measure the hardness from (often secondarily) phase transformed materials, as embedded in the original material. They do not characterize the pristine materials! Clearly, one needs nano, micro and macro depth-sensing indentation in addition to the technical ones, for judging the materials mechanics. Most of the time, materials are not under such very high pressure. Also, the primary transition onset is important for material's mechanics (e.g. failure or fatigue).

The comparison of hardness measurements of sodium chloride is particularly revealing, because (as in the case of sapphire and soda-lime-glass) two consecutive phase transitions are involved. The



**Figure 1:** Kaupp-plot ( $F_N$  versus  $h^{3/2}$ ) of published depth-sensing macro-indentations [5] showing kink-point intersections (phase transformation onsets) and linearity up to 80 N load; an " $h^2$  relation" as believed in ref. [7] is excluded; the data have been extracted from ref. [7]; the correlation coefficients for all six regression lines are  $r > 0.999$ .

literature knows Vickers microhardness data from the list for NaCl properties of the MaTecK-Material-Technologie and Kristalle GmbH collection (Jülich, Germany), reporting 0.20 GPa. Probably, this is the same value as cited [7], but  $F_{\max}/h_{\max}$  is not known.  $H_{\text{ISO}}=0.252$  GPa ( $\pm 2\%$ ) was recently measured at  $F_{\max}=10$  mN, but uncorrected after its fcc-bcc phase transition (from the loading data for ref. [8]). After energy and exponent correction before the phase transition onset this gives with  $0.8 k=5.8229$  the  $H_{\text{phys}}$  value of  $0.2376$  mN/ $\mu\text{m}^{3/2}$  (GPa  $\mu\text{m}^{1/2}$ ) (not violating the energy law etc.), as calculated with eqn. (1) [3].  $H_{\text{phys}}$  is only obtained by linear regression of original data pairs without any of the iterations for  $H_{\text{ISO}}$ . The nanoindentation up to 10 mN load (sharp Berkovich,  $1.17 \mu\text{m}$  depth at 10 mN force) creates the halite to cesium chloride type phase transition (fcc to bcc) with onset at  $0.697 \mu\text{m}$  and  $4.233$  mN load. It requires  $+0.04418 \mu\text{J}/\mu\text{N}$  phase transition work [8]. Ref. [8] reports also the activation energy (23 to  $400^\circ\text{C}$ ) of this first transition. The preferred hydrostatic transition pressure is known as  $26.8$  GPa [9]. The calculated second phase transition (bcc to layered CrB-type NaCl space group Cmcm) is hydrostatically expected at  $322$  GPa, metallic from  $584$  GPa [10]. Most probably, the second transition corresponds with the kink in the Kaupp-plot at  $2.49$  N load and  $21.1 \mu\text{m}$  depth [8] according to the loading curve of ref. [7]. The transition work is  $+3.647 \mu\text{J}/\mu\text{N}$ , which is very large when compared to  $+0.04418$  for fcc to bcc of NaCl, or for example  $+0.007066$  for  $\text{SrTiO}_3$ , or  $-0.01126 \mu\text{J}/\mu\text{N}$  for  $\alpha\text{-SiO}_2$  [8]. The discontinuity at  $21.31 \mu\text{m}$  depth of a sharp Vickers is a candidate for the predicted Cmcm phase of NaCl.

### Vickers hardness test and other one-point-load macro-hardness tests

The load for HV varies in three ranges from  $0.1$  N up to  $1500$  N (HBW10/3000 even with  $30000$  N; the W indicates tungsten-carbide); the normal range is  $40 - 980$  N (HV4 - HV98). The Vickers hardness test is most similar to the pyramidal instrumental depth-sensing, as the Vickers indenter can be used in both techniques. One indents to the chosen load, holds for  $10-15$  s (now  $14$  s), unloads, and calculates HV from the average of the diagonals  $d$  of the impression. The standard is given by eqn. (3), where  $F_N$  is in kpf (kilopond force, it is a very old standard) and  $d$  the diagonal length in mm of the residual impression. Then, after conversion to  $\text{N}/\text{mm}^2$  units one reports (m HVn), where m is the hardness value and n the vertical load  $F_N$  in kpf.

$$\text{HV} = 1.86 F_N / d^2 \quad (3)$$

The first flaw deals with the dimensional error. Since the depth  $h$  is geometrically related with HV's impression diagonal  $d$  according to eqn. (4), the  $d^2$  relates again with  $h^2$  rather than with  $h^{3/2}$ . This gives again a faulty inherent " $F_N \propto h^2$ " relation (instead of the physically deduced eqn. (2)), as in the instrumented depth-sensing force-depth curve [2,3].

$$h = d / (2\sqrt{2} \tan 68) = d / 7.006 \quad (4)$$

Next to the dimensional violation there is the second flaw: violation of the basic energy law. The applied load is not only used for the indentation depth but with  $20\%$  force and thus energy loss (physically correct  $h^{3/2}$ ): the sum of stress formation and plasticization, including sink-in or pile-up, requires energy (if correction of  $h^2$  to  $h^{3/2}$  is not performed, the energy loss would be  $33\%$ ) [1,3]. Long-range features, often with pile-up around the square impressions, have long been seen. Their universally quantitative occurrence in addition to the created stress ( $W_{\text{longrange}}$ ) derives from the physically deduced ratio of the different work contributions in eqn. (5) [1]. Clearly, the non-consideration of pressurizing and plasticization work is violation of the

most basic energy law! The same is true with HB, HR, Shore, rebound, and the techniques that use spherical impression instead of pyramidal/conical ones, because these must also obey the physical relation of eqn. (2) ( $h^{3/2}$  instead of  $h^2$ ) as quantitatively deduced for depth-sensing indentation. Also the UCI-Vickers hardness values, using ultrasound frequency, suffer from the same flaws.

$$W_{\text{applied}} / W_{\text{indent}} / W_{\text{longrange}} = 5/4/1 \quad (5)$$

The third flaw is even more severe than in depth-sensing indentation, because the forces/works and depths are much larger (compare the NaCl, sapphire, and soda-lime-glass cases in Figure 1. Inevitably, there must be several endothermic or exothermic phase changes following each other, not to speak of hidden horizontal cracks that can also occur upon pressure release at the unloading. Furthermore, one-point measurements (rather than linear regression of loading curves with Kaupp-plot) bare the risk of uncontrolled errors. This fact makes it difficult to judge the reliability of HV etc. measurements that could in principle be corrected for energy law and dimension (requiring depth with tip rounding correction), but not with respect to force dependent phase transformations under pressure, the detection of which require analyzed force/depth curves with the physically founded exponent  $3/2$  on the depth (eqn. 2) [2,5].

The interpretation difficulties are demonstrated for HV measurements with the test material 316L stainless steel. The general claim is that HV values must not depend on the load. A publication of 2016 gives a value of  $281.6$  HV0.1 ( $\text{N}/\text{mm}^2$ ) at  $0.981$  N load [11]. The rounding of the Vickers pyramid is not given (its influence is eliminated by comparison with test-plates impressions), but we calculate for ideal Vickers a depth of  $3.66 \mu\text{m}$ . Another publication of 2016 reports  $280$  HV3 ( $\text{N}/\text{mm}^2$ ) at  $29.43$  N load [12] at a calculated depth of  $20.12 \mu\text{m}$ . Important questions are: why is the value for the much deeper and  $30$  times higher force smaller by  $1.6$  MPa? Could it be experimental error (this is calibration at a test material!), or was the tip rounding too different, or are there undetected cracks, or are consecutive phase transitions at the  $30$  times higher force exothermic? Such considerations are missing, but the  $208$  HV3 value was also converted into  $217$  HB;  $95$  HRBmax, and  $89$  HRB. Numerous calibration tables exist and equipment software often displays such converted results as well. The most important of the conversion formulas that interconnect the various techniques are listed in Table 2. Such conversions are termed "approximate" (conversion norm:  $\pm 3.5\%$  of HV), but their use indicates their correlation. That means: all of the single-point macro-indentations exhibit the same flaws with respect to physics, notwithstanding the apparent technical problems. Clearly, size effects due to phase changes are assumed to stay within the large error allowance, and the end radii of the Vickers and Knoop pyramids are not taken particular care of. Apparently, Table 2 is only valid for the same force, and these techniques are by no means universal, but they need for every material a separate test sample with "known" HV that must have been agreed upon. The phase change events are not considered and neither can they be detected by the 1:1 calibration, even though the forces vary from  $0.1$  to  $150$  kpf. Conversely, depth-sensing is universally applicable to all solid materials but requires knowledge of

|           |                                    |
|-----------|------------------------------------|
| HV to HB  | HV $\approx$ 1.05 HB               |
| HV to HK  | HV $\approx$ HK (low load region)  |
| HB to HV  | HB $\approx$ 0.95 HV               |
| HRB to HB | HRB $\approx$ 176-1165/HB $^{1/2}$ |
| HRC to HV | HRC $\approx$ 116-1500/HV $^{1/2}$ |

Table 2: Some conversion formulas for one-point-load hardness values.

the tip rounding that should be small enough, so that its influence can be treated and corrected for as initial effect. ISO uses iterative relation to a standard like fused quartz or aluminum. However, the physically founded depth-sensing obtains absolute hardness values without test samples [2,3], and it detects phase transitions directly as in Figure 1 and [5].

These considerations clearly indicate the close relation of the empirical single-point load macro-techniques that use either surface (HV, HK, HB) or depth measurements (HR, Shore, etc.) to the instrumental nano- and micro- and macro-indentations. Thus the same flaws, as in the ISO standards or FE-simulations, as based on the Oliver-Pharr technique, are involved: first the violation of the basic energy law, second the wrong dimensional error (violation of the Equation 2), and third the non-consideration of phase transformations that cannot be detected during the load and hold periods. The high load capabilities of depth sensing should be extended above 80 N. Clearly, depth-sensing measurements should always be separately available for every material's charge in addition to the fast HV, HR, etc. measurements for rapid and on-site production control, in order to avoid risks from unrespected phase change onsets giving polymorphs with different mechanical properties. And the study of crack onsets is important.

### Tension, compression and speed of ultrasound for Young's modulus $E$

Elastic moduli cannot be obtained by one-point-load hardness tests, but ISO iterates it with depth-sensing unloading. The transformation of  $E_{r-ISO}$  into "Young's"  $E_{ISO}$  from exhaustively iterated unloading curves is achieved with eqn. (6), where both the Poisson's ratio and modulus of the material and the indenter (diamond) occur. This gives values with unchanged dimension but still burdened with the violating of physical laws by the three major physical flaws (dimensional, energetics, unclear solid phase). ISO calls such  $E$  values from unloading curves "Young's" moduli.

There may however be severe objections against equating indentation moduli  $E_{ISO}$  with Hook's Young's moduli. This holds also for the indentation  $E_{r-phys}$  and with eqn. (6)  $E_{phys}$  (the pendant to  $H_{phys}$ ) [3] with different dimension ( $\text{GPa } \mu\text{m}^{1/2}$ ) [7].

$$1/E_r = (1-\nu^2)/E + (1-\nu_i^2)/E_i \quad (6)$$

It does not help that the UCI-Vickers hardness test uses ultrasound response, which requires an effective elastic modulus  $E_{eff}$  from calibration tables for consideration of the E-module. UCI is not a technique for modulus measurement. The reason for eqn. (7) is the universal eqns. (2) and (5) for indentations, which means long-range work for pressurizing and plasticization consumes 20% of the applied work, and thus force, in case of correct dimension according to eqn. (2) (or 33% as long as the false exponent 2 on  $h$  would be applied). But the use of  $E_{phys}$  requires some efforts with the calculation of the initial slope of the unloading curve using the original data, rather than a ruler to the recorded curve.

$$\text{Indentation-}E_{r-phys} = 0.8 S / (2h_{max}^{1/2} \tan \alpha) \quad (7)$$

In the absence of original data it can appear impossible to graphically approach the initial slope  $\Delta F_{max} / \Delta h_{max}$  that is the iteration result by Oliver-Pharr. It claimed  $E_{ISO}$  value of 73 GPa (Berkovich,  $R=50$  nm) from ref. [6] up to 215.8 nm followed by creep up to 266 nm depth. Actually, ISO iterates  $A$  (projected contact area) with an unrelated standard for final height  $h_f = h_{max} - 2F_{max} / S$  for  $A$  and

fits 80% or 50% of the exponential unloading curve iteratively with  $F_N = A(h_{max} - h_{final})^m$ , where  $A$ ,  $h_{final}$ , and exponent  $m$  (between 1 and 3) are the free parameters. Stiffness  $S$  at peak load is then obtained by the differentiation  $dF_N/dh = S = Am(h_{max} - h_{final})^{(m-1)}$  for obtaining the maximal slope. This circumvents the slope detection.  $E_{r-ISO}$  is then calculated as  $\pi^{1/2} S / 2 A_{hc}^{1/2}$  and the result is called "Young's modulus" after application of eqn. (6). This is objectionable ISO standard.

The principal problem with such definition of an indentation modulus is the anisotropy of most materials that cannot be tackled by indentation, irrespective of the possible physical corrections eqn. (7). For example, it is known from the fact that different faces of a crystal give different  $E_{r-ISO}$  moduli depending on the different predominance of the crystal faces towards the tip (e.g.  $\alpha$ -SiO<sub>2</sub> varies  $E_{r-ISO}$  between 105.0 and 133.6 GPa onto 5 different faces) [13]. The skew indenter surfaces collect in fact a mixture of some sort of different elastic moduli from all of the different directions around the tip and there are also shear-moduli involved upon the unloading. This is far away from unidirectional Young's modulus, depending on Hook's law eqns. (8) and (9). Thus,  $E_{ISO}$  is incompatible with Hook's law, and indentation- $E_{phys}$  can also not be made compatible. Any similarities of  $E_{ISO}$  values with Young's moduli are thus fortuitous. They derive from the iterative fitting to the unidirectional Hook's value of a standard. They are therefore fortuitous, because of both the multi-directionality and because of the striking physical errors of  $E_{ISO}$ . They do not have the same meaning, as might be suggested by the unfortunate common wording. Fortunately, an extensive amount of well-studied Hook's Young's moduli for all independent directions of preferably cubic and other high symmetry crystals are tabulated and do not need repetition by indentation. The complexity of the 6x6-matrix treatment of Young's moduli, leading by some matrix symmetry to generally 21 independent elastic constants that are further reduced by crystal-symmetry to 9, 7, 6 and in the cubic case 3 independent moduli has been amply described (for example in ref. [14]). So it is suggested to call  $E_{phys}$  eqns. (6) and (7) "indentation modulus" and check, whether the three-dimensional bulk modulus, as obtainable from hydrostatic pressurizing, is an equal or superior parameter for characterizing the elastic properties of micro or macro materials.

It is essential now to briefly repeat the Hook's technique for obtaining Young's moduli  $E$ , where the shear modulus detection is excluded. The clearest experimental determinations of  $E$  are by tension/compression eqn. (8) or ultrasound speed eqn. (9). The uniaxial tension or compression gives the simple elongation/depression Hook's law eqn. (8), as long as these are fully reversible. Transversal thinning/thickening is always mentioned, but transversal work can apparently be neglected.  $L$  is length,  $p$  is the generated pressure (force per area),  $E$  Young's modulus.

$$\Delta L / L = p / E \quad (8)$$

Eqn. (9) recalls the ultrasound speed technique in long rods with diameters smaller than the ultrasound-wavelength, excluding shear-waves, where frictional loss may be small or ineffective. It is used for the longitudinal speed  $v_s$  in such rods, where  $E$  is Young's modulus and  $\rho$  is density. These and more complicated Hook's techniques are generally accepted textbook physics.

$$v_s = E^{1/2} / \rho^{1/2} \quad (9)$$

For practical reasons we regret that the Hook's law techniques require much larger test samples with highly specialized geometric shape. They are therefore more difficult to perform and less versatile than would be indentations, that appear however inappropriate for  $E$ .

The present situation is at best exemplified with the simplest case, cubic isotropic aluminium.

We have to distinguish tabulated Young's modulus ( $E=69$  GPa), shear modulus ( $G=25.5$  GPa) and bulk modulus at hydrostatic compression ( $K=76$  GPa). This compares to claimed invalid  $E_{\text{ISO}}=73$  GPa [6] that must be decreased to 10.7 GPa by making the physical corrections. Clearly, nothing from the unloading is fitting with the reliable Hook's values. There is no hope left that indent- $E_{\text{phys}}$  (mN/ $\mu\text{m}^{3/2}$ ) values could be converted into Hook- $E_{\text{phys}}$  (mN/ $\mu\text{m}^2$ ) values (for example by division with  $h_{\text{max}}^{-1/2}$ ), because they would have totally different meaning. Again, it does not help that  $E_{\text{ISO}}$  is iteratively fitted with respect to a unidirectional Hook's Young's modulus of a test material.

The consequences for the recent use of physically unsound  $E_{\text{ISO}}$  values are detrimental, when their use for mechanical parameters is considered. The particular importance of an indentation modulus is evident from numerous applications. The listing 1 through 12 indicates various examples.

1. All elastic properties
2. Input for FE-simulations
3. Stress-strain response
4. Film hardness and film adhesive strength
5. Strain hardening
6. Creep calculation
7. Material fatigue, fatigue strength
8. Adhesion calculation (DMT or JKR)
9. Viscoelasticity studies
10. Sliding friction coefficient
11. Contact area at dynamic testing in continuous stiffness mode
12. Fracture toughness

At present it appears only possible to calculate Young's modulus  $E$  of new materials for certain directions and test the quality of such calculations with as close as possible materials, for which the Hook's values are known, or to rely on indentation- $E_{\text{phys}}$  or on bulk modulus  $K$  by hydrostatic pressure experiments for the consideration of reliable elastic materials properties.

### Reasons for obeying physical laws

It is very clear that mechanical properties must not violate basic physics, be it in academia, industry, medicine, or daily life. That does not mean that purely empiric methods like the Mohs hardness scale (who scratches whom) are also useful. However unphysical parameters must not violate physics. And one must not try to make physical correlations with unphysical parameters. For example, Mohs says steel cuts leather. However, there is also mechanochemistry that explains why barbers can sharpen their blades with leather [15]. Clearly, also the size of the components and the chemical composition of the solids play an important role (here polymers are tribomaterials) [15]. Brittleness, ductility, lubrication are further qualities apart from hardness and elasticity, that have their meaning in particular applications. Hardness and elastic moduli should be physical rather than empirical due to countless technical constructions where different materials must work together and alloys or composites must be compatible rather than fail

upon short use. Materials are often used under low pressure where they are not phase-transformed. And different materials have their phase change onsets at varied pressures. This provides severe risks when they are perhaps only compatible under very high pressure as high pressure polymorphs, but not at lower or ambient pressure where they are at rest. Everyone knows that virtually all purchased goods with granted guarantee periods fail (shortly) after that period, or airliners must have very short control and replacement terms of all parts, because they must not fail. Only physically sound parameters of hardness and modulus with all of the numerous other mechanical parameters that depend on them should be used, instead of violating basic physical laws with  $H_{\text{ISO}}$  and  $E_{\text{ISO}}$ . The dilemma of ISO-standards against physics is a thread for daily life, because falsely calculated materials bear enormous risks for lifetime and failure. Some examples are composite materials (also solders) that may not properly fit together, or exploding turbines, or breaking windmill blades, or micro-cracks in airplanes and huge pressure vessels of power plants, or breaking medicinal bone implants due to incompatibility, etc.

### Conclusion

The comparison of single-point load macro-indentations with physical and mathematical precisely handled depth-sensing nano, micro, and macro indentations reveals three major flaws of the former that can be and have been removed for the latter [3]. All depends on the physically deduced exponential law  $F_N \propto h^{3/2}$ , instead of the believed  $h^2$  from Sneddon, Oliver-Pharr, and ISO standards [3]. The same flaws (violation of the basic energy law, dimensional error against physics, and disregard of phase changes under load) are also inherent in present ISO and ASTM standards that still do not apply basic physics from the depth-sensing techniques. Since the one-point force techniques are much more rapid and comfortable in industry, these purely empiric techniques with standardized calibration necessities at test plates and tables for different material types are now only acceptable, when the materials in question have also been studied on the genuine physical basis with force/depth curves, as described here and in ref. [3]. Depth-sensing ISO-standards are subject to urgent changes for complying with physics. Most serious in view of failure risks are the present disregard of phase transition (phase change) onsets, and size depending very large differences between faulty  $H_{\text{ISO}}$  and the much more precise  $H_{\text{phys}}$  values with different dimensions. Similarly, indentation elastic modulus  $E_{\text{ISO}}$  (falsely called "Young's modulus") fails: it suffers from the same physical flaws and has no relation to unidirectional Hook's law. The unloading skew pyramid or cone surfaces collect a mixture of multidirectional elastic moduli and shear moduli. Therefore, indentation-moduli have a totally different meaning than Hook's Young's modulus. They cannot be given the same name, and the term  $E_{\text{ISO}}$  is also worthless due to three physical flaws, and to questionable iterating fitting techniques as initiated by Oliver-Pharr and taken up by ISO. The incredible claim that ISO would deal with unidirectional Young's modulus has to be rejected. It is not at all available for indentation unloading.  $E_{\text{ISO}}$  and deductions there from are unphysical and their use must be discontinued. The use of indentation- $E_{\text{phys}}$  or bulk moduli  $K$  should be used in situations where the one or the other appears more appropriate or better both for the mechanical characterization of materials. Phase changes under pressure must be controlled as detected from the mathematical analysis of instrumented loading curves, so that the rapid single point high-load indentations can find the appropriate interpretation.

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