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Dilemma between Physics and ISO Elastic Indentation Modulus

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Abstract

This paper challenges the ISO standard 14577 that determines the elastic indentation modulus by violating the first energy law, and omitting easily detected phase change onsets as well as initial surface effects under load. The double iteration for incorrect fitting indentation modulus to Hook's law Young's modulus of a standard with up to 11 free parameters must be cancelled and discontinued. The iterative evaluation of the elastic modulus $E_{r\text{-ISO}}$ can by far not be reproduced by iteration-free direct calculation of E_r, when using the underlying formulas for S, h_c, A_{hc}, and ε. For cubic aluminium the divergence amounts to a factor of 3.5 or 3.1, respectively (both smaller for the non-iterated calculations). Every interpretation of indentation moduli as single unidirectional "Young's moduli" is false. They are mixtures from all directions and include shear moduli. The three different packing diagrams of body centered cubic α-iron exemplify the mixture of three independent Young's moduli (and thus also three shear moduli) even in this simple but already anisotropic case. More linear moduli ensue in lower symmetry crystals as exemplified with α-quartz. The first physical indentation modulus is deduced by removal of the physical errors of $E_{r\text{-ISO}}$, or after indenter compliance correction E_{ISO}. E_{phys} does no longer violate the energy law. Five face-dependent elastic indentation moduli of α-quartz at the obsolete $E_{r\text{-ISO}}$ level and two tensional Hook-law Young's moduli are compared with all of its six resonance ultrasound spectroscopy (RUS) evaluated Young's moduli, and with the bulk modulus. The dilemma between ISO and physics is particularly detrimental, as E_{ISO} is used for the calculation of very frequently applied mechanical parameters. These propagate the errors into failure risks of falsely calculated materials with severe violation of the basic energy law and other physical laws for daily life. Difficulties with the urgent settlement by new ISO standards are discussed. First suggestions for the use of E_{phys}, or S_{phys}, or eventually measured bulk modulus K are made. This should be urgently evaluated and discussed.

Keywords: Bulk modulus; Compressibility; Data correction; Errors of elastic moduli; Falsely calculated materials; Falsified iterations; Hook-RUS-technique; Indentation modulus; ISO-standard; Mechanical parameters; Physical modulus; Shear modulus; Young' s modulus

Introduction

The recent correction of the ISO-standard 14577 for indentation hardness (H_{ISO}) and so-called Young's modulus (E_{ISO} from $E_{r\text{-ISO}}$) [1-3] enabled the availability of physically correct mechanical quantities. This implied return to the first energy conservation law after half a century, removal of dimensional errors. Furthermore, any of the occurring surface effects and phase transition onsets under the mechanical load is now revealed upon depth sensing (not available to single load techniques like Vickers, Brinell, Rockwell, etc. hardness), and they can be avoided at lower load [1-3]. Iterations and approximations are now easily avoided at the expense of linear regression analyses. Indentation hardness H_{phys} was deduced as physical quantity for the first time and ISO modulus definition was provisionally improved. Materials can now be physically correct described and numerous unexpected applications ensue by use of simple closed formulas. But there remains further trouble with ISO-modulus E_{ISO} [2,4]. It is falsely called "Young's modulus" but a unique indentation modulus E_r as compared to unidirectional Young's modulus E, shear modulus G and bulk modulus K. It is therefore timely to unravel the misleading situation with $E_{r\text{-ISO}}$ and E_{ISO}.

Materials and Methods

The nano-indentations used a fully calibrated Hysitron Inc. Triboscope(R) instrument with AFM leveling in force controlled mode with a Berkovich indenter (R=110 nm) at the exclusion of phase change below F_{max} and validity check with the "Kaupp-plot" F_N versus $h^{3/2}$ [2,5] throughout, also for correction of initial surface effects. Stiffness values S are calculated by linear regression of the upper unloading data points, as long as these decrease linearly. Crystal packing was imaged by use of the program Schakal 99 from Egbert Keller, University of Freiburg i.Br., Germany. The cited literature data have been checked and interpreted in view of the mathematically deduced new general physical laws with closed simple formulas in accordance with validated experimental data, excluding all forms of iterations or approximations. Phase changes under load were detected by kink-type discontinuity in linear regressions. The precise intersection point was obtained by equating the regression lines before and after the onset of the phase change. The necessary energy law correction by virtue of the physically deduced $F_N \propto h^{3/2}$ law is 0.8 [3] (the energy law violation correction of ISO would be 2/3, as long as the unphysical exponent 2 on the depth h would still be continued).

Results and Discussion

Flaws of the ISO indentation modulus

The reduced ISO indentation moduli values are defined as $E_{r\text{-ISO}} = S\,\pi^{1/2}/2\,A_{hc}^{1/2}$ and iteratively obtained against a standard. These are therefore no absolute but relative quantities. The corresponding definition of absolute E_r is then $S\,\pi^{1/2}/2\,A_{projected}^{1/2}$. At first, ISO iterates the unloading curve according to $F_N = B(h_{max} - h_{final})^m$ with the three independent parameters B, h_{final}, and exponent m for obtaining the

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maximal slope with $dF_N/dh=S=Bm(h_{max}-h_{final})^{(m-1)}$. $E_{r\text{-ISO}}$ is then calculated as $S\,\pi^{1/2}/2\,A_{hc}^{\;1/2}$ and from there E_{ISO} with $1/E_r=(1-\nu^2)/E+(1-\nu_i^{\,2})/E_i$, where i denotes the values of diamond. It follows a further iteration of A_{hc} with eight parameters C_n (also sign change option allowed): $A_{hc}=\pi E_r^{-2}(C-C_f)^{-2}/4=(24.5h_c^{\,2}+C_1h_c+C_2h_c^{\,1/2}+C_3h_c^{\,1/4}+\cdots+C_8h_c^{\,1/128})$ for fit to the Young's modulus of a standard. This result is then falsely called "Young's modulus" [6] in ISO-14577. This iterative procedure with eleven free parameters does however not obtain a physical quantity [2]. It is per se troublesome.

Even more serious problems occur with the convergence prescription of the iteration. While the direct calculation of A_{hc} is possible with $S=dF_{max}/dh$ and the deduced formulas (11, 12, 17) of [6], this path was not followed by ISO, but they standardize the described iterative procedures by fitting against a standard. The diversion between the two paths is enormous. By using the direct path we follow the defined underlying basis of [6] and obtain for the unloading curve of, for example, cubic aluminium [7] with uncorrected $S=dF_{max}/dh=902\cdot10^3/35.79$ nN/nm, and $\varepsilon=0.72$ the value $h_c=h_{max}-\varepsilon F_{N\text{-}max}/S=190.03$ nm and the value of $E_{r\text{-ISO-}Ahc\text{-direct}}=23.74$ nN/nm^2. When the direct calculation with A_{hc} is changed for $A_{projected}$, we obtain the absolute $E_{r\text{-direct}}=20.9$ nN/nm^2. The respective error factors 3.1 and 3.5 when compared with $E_{r\text{-iso-iterated}}=73$ GPa [7] are enormous! They falsify convincingly these ISO iteration standards not only for this example. Such discrepancy similarly happens with other materials but it can be less drastically. The described iteration procedures for $E_{r\text{-ISO}}$ cannot describe the claimed above definition of $E_{r\text{-ISO}}$. This demonstrates enormous data-treatment by false iterative fitting to unrelated Young's modulus.

Clearly, the "Young's modulus" claim of ISO is faulty from the beginning. It cannot describe a response to a unique linear elastic stress. Indentation moduli are face-dependent multiple mixtures of linear and shear moduli around the skew conical, pyramidal, spherical, and further indenters. Furthermore, it violates the energy law because F_N creates not only work for volume but also 20% of its value work for pressure generation and long-range modifications. This surprising generality has been easily deduced [1-3,8]. Finally, ISO does not detect and avoid any phase transition onset that might occur at $< F_{max}$ [1-3], which must be done by checking for sharp kink in the linear Kaupp plot [1-2,5] of the loading curve.

The physical indentation modulus

With the generally required aim for minimal change of existing hypotheses we start with the formal relation between unloading stiffness ($dF_{N\text{-}max}/dh=S$; experimental) and elastic modulus (E_r) in the form of $E_r=\pi^{1/2}S/2\,A^{1/2}$ as above, which appears to have been successful in several Russian papers of the 1970s and 1980s, as cited [6]. The $\pi^{1/2}/2\,A^{1/2}$ factor is obviously derived from elastic contact theory arguments. The $A^{1/2}$ reflects one-dimensionality. It was adopted by [6] and ISO with the complication that it had been termed as root of contact area $A_{hc}^{\;1/2}$ (see preceding paragraph). But S must again be corrected so that $S_{phys}=0.8\,S$, because the so corrected F_{Nmax} (after initial surface effect correction and at < onset of phase change) is it's constituent in the form of ΔF_{max}. A dimensional correction as required for H_{phys} [3,8] is not required, as the Δh constituent of S is only related to the unloading curve, according to this definition of E_r. As already outlined in the previous paragraph: indentation moduli are not "Young's moduli".

The physical formula on that basis after the shortening out of $\pi^{1/2}$ is thus $E_{r\text{-phys}}=0.8\,S/2\,h_{max}\tan\alpha$ (nN/nm^2) [2], and it avoids energy law violation, phase change onset exclusion at $<F_{max}$, and initial surface effects. All what's needed is the simple mathematic correction after linear regression of the loading curve before the kink. $E_{r\text{-phys}}$ also avoids multi-parameter iteration fitting to a standard's Young's modulus. We obtain the absolute elasticity modulus of $E_{r\text{-phys}}=16.73$ nN/nm^2 for aluminium. That is very different from the obsolete iterated ISO-modulus (73 GPa) published [7].

Comparison of indentation with Young's moduli

We must now stress the principal difference of indentation moduli $E_{r\text{-phys}}$ and unidirectional Young's moduli. Valid Young's moduli detections require Hook's law, for example by unidirectional reversible tension ($\Delta L/L=p/E$; p is pressure), or ultrasound speed in long rods ($v_s=E^{1/2}/\rho^{1/2}$; ρ is density). In more complicated cases resonance ultrasound spectroscopy (RUS) is used. Correct linear Young's moduli are unique in different directions, excluding shear-moduli. The 6 by 6 matrix of Young's moduli gives by cancellation 21 of them. This decreases further by crystal symmetry to 9, 7, 6, or in the cubic case 3 independent moduli, as is generally communicated. Conversely, indentation moduli are multiple mixtures of linear and shear moduli around the skew conical, pyramidal or spherical and further indenters from all sides. They are face-dependent due to their different weight at different positions. As there seems to be some uncertainty about isotropy of cubic crystals that have been termed as "very isotropic" for the case of metals [6] and also by ISO, we demonstrate here cubic anisotropy. Figure 1 exemplifies the different packing of bcc α-iron along [100], [110], and [111]. These directions exhibit marked different packing properties and thus also three independent linear moduli in these directions, according to the complete matrix analysis. This is a basic model for all types of cubic crystals as for example fcc aluminium or sodium chloride, etc. Furthermore, also three independent shear moduli ensue upon indentation into cubic crystals. The situation becomes more complicated in all other crystal systems with more elastic constants. Importantly, Figure 1 indicates that the common relations between Young's, shear, bulk modulus, and Poisson's ratio cannot be applied to any crystalline materials, due to their anisotropy. However, that has been frequently carried out.

A more complex system is exemplified with trigonal-trapezoidal α-quartz ($P3_121$ or enantiomer $P3_221$) that mixes 6 independent Young's moduli upon indentation, according to the matrix analysis; each with additional shear moduli. The dilemma is evident from Figure 2. The various reported moduli values are reported by Crystan Ltd [9] and the linear moduli were determined by NIST with the elaborate RUS technique [10]. Only the −18 value is still judged "troublesome". Also the tensional moduli E for two directions and the shear modulus G from bending shearing of the main axis and the hydrostatic bulk modulus K [9] are also included. These values are compared with the

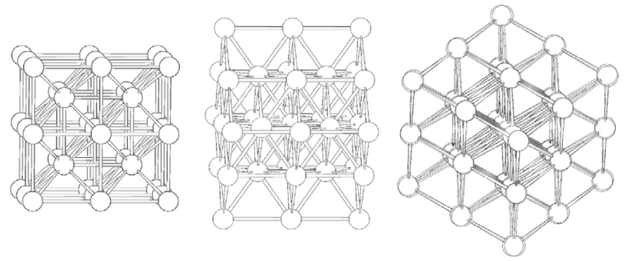

Figure 1: The packing variation at the bcc α-iron (Im-3m; a=2.8665 Å) along the frontal [100], [110], and [111] directions, from left to right, respectively, showing the variable packing differences in these directions.

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obsolete phase-transformed iterated ISO indentation E_{ISO} (no surface designation and no original data available [6]), the for that purpose still useful though obsolete phase-transformed iterated E_{r-ISO} moduli on 5 different faces of α-quartz from 2005 [11], and the formula for the physical indentation modulus [2].

The largest variations are in the Hook RUS Young's moduli series. The main axis tensile values are closest to the highest RUS values. All of these and the shear and bulk moduli are much smaller and unrelated to the much higher obsolete indentation modulus of Oliver-Pharr who initiated the ISO iterative modulus determinations with the false claim that these be "Young's moduli" [6]. Similarly, our five old indentation modulus values on five different faces [11] are much too high at the obsolete ISO iteration level, due to the faulty iterations and phase transition. Unlike the strong variation in the RUS series, they vary within 20% (the largest at the direction with the thinnest channels), indicating at least the incompatibility and the surface dependence. However, these experimental values [11] are now obsolete. Valid E_{phys} indentation moduli appear most promising for the correction of the further mechanical parameters that derive from indentation (Figure 3). They can not be identical with bulk moduli K. But K-values from compressibility measurements are much more difficult to obtain and their use with respect to indentation data would have to be carefully discussed. But this might perhaps also appear promising, because K includes all types of elasticity. Again, any relation of indentation moduli to Young's moduli is excluded and must not be tried.

Modulus-containing mechanical parameters

Figure 2 indicates that the choice of an elastic modulus for the characterizing of further mechanical properties is not yet clear when most easily obtained indentation results are involved. The present problems are detrimental because of the numerous deduced parameters. The widespread use of the iterated so called "Young's modulus" indentation moduli E_{ISO} must be stopped. Figure 3 collects the still most frequently applied uses of false-designated obsolete iterated E_{ISO} with its numerous described flaws. This is a detrimental situation with high general risk, as these values are unphysical. Any unsuitable choice for elasticity deduced mechanical parameters is detrimental. The dilemma of ISO-standard 14577 with physics has to be replaced as soon as possible for the sake of correct science and even more importantly for every day's security; because most of the mechanical parameters in Figure 3 are ill-calculated against basic physics and falsified iteration. The perhaps first ray of hope for a change is perhaps the use of the bulk (volume) modulus K in the rheological Kelvin-Voight model [12,13]. It should be noted in this respect that the parameters containing the H/E fraction change their dimension with E_{r-phys} or with K. This might pose difficulties with their meanings. With the other mechanical parameters of Figure 3 only their size will strongly change. The iterated ISO-moduli are obsolete and the non-iterated ISO moduli are still burdened with the physical flaws. But energetic and phase integrity flaws of the latter can be solved for reaching E_{phys}. The present situation is still involved. Detailed discussion and much work must resolve these most important questions.

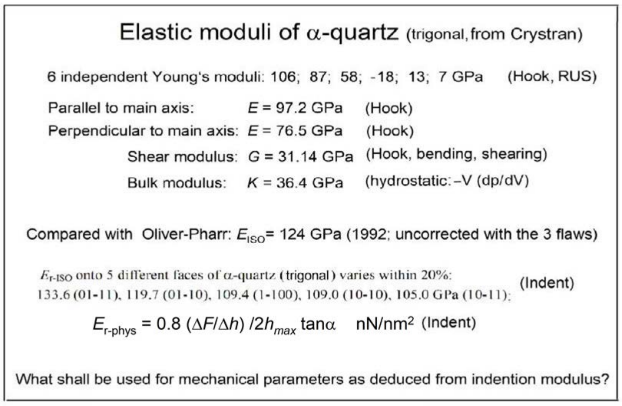

Figure 2: Comparison of various elastic moduli of α-quartz.

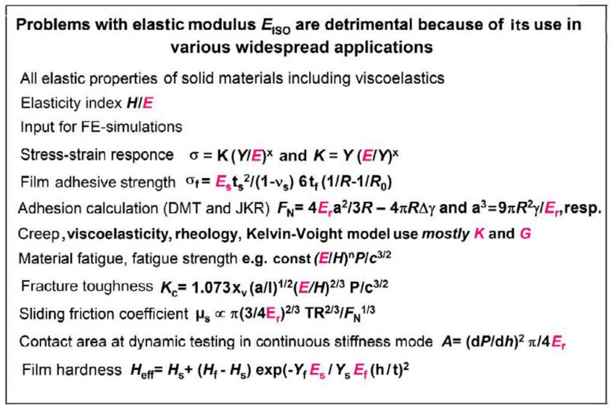

Figure 3: Some applications of elastic modulus as deduced for mechanical properties.

Conclusion

The situation of elastic modulus from depth sensing indentations requires complete revision. The physical flaws of E_{ISO} are energy law violation, not caring for exclusion of phase change onsets, and not correcting for initial surface effects under load. Another very severe flaw derives from falsifying iterations with up to 11 free parameters (free sign change option) by obviously converging to Hook-law Young's modulus of standard materials by misinterpretation of the meaning. However, the indentation experiment is not at all unidirectional but contains linear and shear contributions from all sides of the skew indenters. This behavior also violates against the underlying definition of the ISO modulus and must be urgently discontinued. The false iteration becomes evident for example from cubic aluminium with $E_{r\text{-ISO-}Ahc\text{-iterative}}$=73 GPa and iterative-free $E_{r\text{-ISO-}Ahc\text{- direct}}$=23.74 nN/nm^2, or $E_{r\text{-absolute-direct-}A\text{projected}}$=20.9 nN/nm^2 (all with the physical faults). Thus only the newly defined absolute E_{phys} of 16.73 nN/nm^2 or the corrected stiffness S_{phys}=0.8 S contain all elastic effects around the tip impression. Indentation moduli are thus not related to Young's moduli. Fortunately, E_{r-phys} or S_{phys} do not contain any of the physical and iterational flaws of $E_{r\text{-ISO(-}Ahc)}$. There remains the question whether E_{r-phys} can be rapidly and broadly applied for the elasticity derived parameters of Figure 3, Alternatives might be S_{phys} or compressively measured bulk modulus K. Such decision may depend on theoretical or practical arguments. Corresponding series of data pairs from both fields for comparison are missing and should be made available for evaluation.

E_{ISO} variations should no longer be used for Figure 3 parameters and the like. Why shouldn't we stay with physics? Who is liable upon failure of ill-calculated materials, and what about the judge and the victims? ISO standardization procedures are slow:

1. We need new ISO standards and new textbooks for indentations!

2. We must be enabled to rely on material's properties and save health, time, and money!
3. Everything must become easier with simple mathematics without fittings and/or iterations!
4. We must no longer violate the first energy law and other basic physical laws!
5. We must honestly teach on basic physics!
6. We must remove previous errors!
7. We must make daily life safer in the future!
8. It is dangerous to fight against experimental evidence and convincing physical deductions based on elementary mathematics!
9. Life becomes safer, and brighter with admission of the physical truth.

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