



Indentations Do Not Provide Elastic Moduli: Inelasticity

Gerd Kaupp*

University of Oldenburg, Chemistry Department, Germany

***Corresponding author:** Gerd Kaupp, University of Oldenburg, Chemistry Department, Germany.**Received Date:** March 20, 2026**Published Date:** April 17, 2026

Abstract

Indentations with pyramidal or conical diamond indenters onto solid materials and their stiffness or moduli are inelastic, because stable impressions are formed. They are different from elastic moduli as obtained from Hook's law or RUS (resonant ultrasound spectroscopy). The value of inelastic stiffness over elastic stiffness is easier available by (nano) indentation. And it is covering both the weak elastic and dangerous strong and sudden inelastic mechanical stress, which might become catastrophic stress via mostly available phase-transitions that facilitate cracking at typical forces. All indentations occur inelastic (incomplete splitting) and their face-transition onset forces are obtained as sharp kink points of physically correct force versus depth $h^{3/2}$ parabolas. The inelastic stiffness of technical materials must be as high as possible, as are the calculable transition energies. Proper materials should therefore be compared and selected at the expense of inferior materials. Only physically correct (force vs. $h^{3/2}$, but not h^2 !) analysed inelastic stiffness before and after phase-transition onset kink(s) allow for the safe search of proper technical materials and for the necessary fight against catastrophic failures. But the correct elastic stiffness (Hook or RUS type) are unsuitable, as well as millions of falsely (h^2 instead of $h^{3/2}$) iterated so-called "elastic moduli" that totally lost and loose the undeniable wealth for optimization. The applications of inelastic stiffness are manifold and some of them are suggested.

Keywords: Correct Indentation; Inelastic Stiffness; Elastic Stiffness; Phase-Transition Onset; Crashing; Airplanes; Windmills; Turbulences; Car Accidents; Railway Tracks; Armor; Bullet-Proof Clothing; Balconies; Bridges; Buildings; Earthquakes...

Introduction

The indentations onto solid materials with pyramidal indenters (Standard: Berkovich) to give stable inverted pyramidal holes are very useful, evidently non-elastic, processes (proper microscopes can image the holes as flat triangles and also as inverted pyramids). Their worldwide broad use from 1992 [1] was taken up by enforcing industrial DIN-ISO-NIST Standard-14577, because it provides most easily and rapidly hardness values (force/area) and stiffness values (D_{force}/D_{depth}). But the undue conversion of the latter into Young's modulus (not considering that only

the latter is unidirectional) is fraudulent. The indentations are clearly multidirectional (Berkovich, 3 times 120°) from inelastic (incomplete splitting type) procedures (not as claimed plastically recovered) and there from stiffness and modulus are therefore also inelastic. But people wanted it as "elastic stiffness" for its comparison with the correctly but difficult obtained elastic Young's modulus and there from "elastic modulus". The second error was therefore a mysterious fraudulent trick for still successfully cheating mankind, by using afterwards an extremely strange so-called

unload and load into the created indentation hole for obtaining “elastic stiffness”. The very long verbose wording by describing it in [1] is hardly understandable. Only finite element (FE) calculations can be extracted. The machine program must use such mentioned calculations at least for the material’s elasticity within the “black-box” program of all purchased nanoindentation instruments. These calculate and draw the corresponding figure for the so-called “elastic modulus”. All of these instruments obviously run a not disclosed program, that can be started for printing a load versus depth unloading and reloading curves, both bent to the lower side unload and reload diagram (F_N vs h), but never being fully down to zero load, while the force value is continuously increasing to the F_{max} value and the depth scale is continuing. The linear slope down to not less than about 70% of the loading value is used (according to the questionable undisclosed technique). But all of that is still within the inverted pyramidal hole of the incomplete splitting. And this has been using by millions and further millions over the world, despite the strange imagination of an indentation into a hole. Such stiffness values are highly questionable. Nevertheless, all mankind (including AI applications) likes to easily run black-boxes of purchased instruments, so that such images “might be somehow imaginable, if almost all colleagues use it without complaint”. The stable indentations holes were often depicted microscopically, but the hardly understandably introduction of the technique by [1] is perhaps too complicated. Also, the present Author unfortunately used his purchased black-box with its Handbook, despite his indentation holes images (direct black triangles or conversion to inverted pyramids). But such dangerous behaviour is now corrected in his publications [2] and [3]: we know it scientifically now.

The strange procedure of [1] reports unloads and reloads into and from the before indented copy able hole. But it is not an experimental result, but only obtained by iterative calculation! Why should such initially linear curve-parts of load/reload into the created hole-image, be a stiffness for modulus? And why is there a π for the transformation of Stiffness into the transfer of stiffness into the Berkovich Standard modulus formula? That instrumental procedure yields repeatedly a force/depth diagram with initial straight lines bent towards lower depths. It does so both for the “unload” (interrupted before reaching the depth scale) and for the “reload” up to the maximal force, while the depth scale still runs on. The initial zero point is always far away and never reached. The maximal steepness D_{force}/D_{depth} value of the unloading-reloading curve is unduly called “elastic stiffness”. It is however at best inelastic pressure relief.

Clearly, we have to distinguish the pressure relief before and after the sharp phase-transition onset force. Before the (with unphysical h^2 unknown) kink point force we have only pressure relief when material and indenter diamond separate. At higher forces beyond the kink point the impression forms a polymorph layer between the compressed material and the diamond tip. At short times for indentations only a thin polymorph layer can be formed. Upon removal of the tip it can select to remain on the material surface or at the diamond surface. It might retain its transformation, or it will

mostly revert back to the initial molecular structure. Such pressure-less reformation might proceed elastically.

More interesting are processes of remaining unchanged. Macroscopic examples are known in geology during very long periods of high pressure where the less stable polymorph might survive. The perhaps most impressive example is calcium carbonate $CaCO_3$ [4]. Both polymorphs, the more stable Calcite (trigonal; R3c, No. 167) and Aragonite (orthorhombic; Pmcn, No. 62) can be found at different geographic locations and are mined in pure form as transparent crystals. Their kink value force ratios of Calcite (100) (after the twinning) over Aragonite (-110) are found at 0.3655 / 0.4086 mN force and the corresponding ratio of the phase-transition energy calculations (formulas in [3]) per μN are 15.99/2.758 W_{trans} per μN that were calculated from these loading curves [4]. This reflects quantitatively the higher stability of the Calcite at ambient temperatures. It is about 6 times higher.

Upon indentation, the release of the tip (after as usual short exposure) only a thin layer of phase-changed polymorph can form between material and the diamond tip. That is the case when the energy upon indentation exceeded the one at the loading kink-point of the compressed material between material and indenter. Upon release of the indenter from its inelastically created hole by retraction, the transformed layer material may either stay with the material or with the indenter diamond. The inelastically produced polymorph layer might then elastically revert to the initial material.

In Table 8.1 of [4] Internet we determined from the loading curves of Kearney and Guilloneau. And the necessary phase transition energy is about 6 times higher for Calcite. The indentation unloading holes might in some cases lose some tiny grains that fall into the hole. Or some liquid formation was observed within the holes of indented solid organic polymers such as images for linear PMMA and PC, or glassy polymers like cross-linked CR39 [2,3,5] Springer. Viscoelasticity solids can be further materials for such behaviours.

The criticised so called “unloading/reloading into indentation holes” is complicated enough for directly being understood and the wish for it being useful for stiffness detection is so great, that all materials science with unphysical enforcing DIN-ISO-NIST-14577 Standards and alternate intelligence AI applications in the Internet and millions of respective publications still believe in it. Even the present Author had initially some problems with it when he falsely used the word “elastic modulus” because all worlds have been using it and want to easily obtain and use “elastic moduli”. He has to apologize for some unfortunate unsuitable expressions but it was always with respect to the false enforcing DIN-ISO-NIST 14577 and [1]. And he criticizes their common risks. But, what the hell? These load/unload curves prove that all of the generated impressions stay inelastic stable, whereas elastic indentation would not give stable impressions. He imaged their inverted pyramids shortly and also after six months [6].

The inelastic indentations are small enough for being accommodated by the created crystal faults and already existing

imperfection can be accommodated. Fortunately, we obtain now the inelastic stiffness and also inelastic moduli from the physically correctly analysed loading curve, so that we do not need questionable to false unloads/reloads into indented holes. And we discuss their much higher worth over their elastic stiffness counterparts.

1. Discussion

It has been known by mathematic deduction since 2017 [7] that pyramidal or conical indentations use exactly 20% of their loading work for pressure to the interface between solid material and indenter diamond. This result follows from the loading curve ($F_N = k h^{3/2}$), ($W_{indent} = 0.4 k h^{5/2}$), ($W_{applied} = 0.5 F_{Nmax} h_{max}$) = $0.5 k h^{3/2} h$, giving $W_{applied} - W_{indent} = 5 - 4$, which is 20% of 5. Such pressure (20%) is released upon unloading.

In case of phase-transition at forces above the kink-point of the loading curve, one may have stable polymorph or return to the original phase after release of pressure. Reversed imaging [6] with electronic microscopes shows-up the inverted pyramidal (or conical) image of the stable impression. Both the indentation and the stiffness (Dforce/Ddepth) and there from deduced “moduli” are thus inelastic. Clearly, elastic Young’s moduli are not available by the easy and fast indentations, and why should we anyhow long for these? It must be inelastic stiffness that counts and it makes (nano) indentations still more valuable. In addition to detecting phase-transitions, the transformation energy of which can be calculated (formulas in [3]). They do not correspond with other very complicated techniques for elastic moduli. Examples for correct elastic moduli from the complicated correct Young’s moduli techniques for comparison with standardised indentation modulus of [1] are available for trigonal rock salt (α -quartz) and they show from 0.5 -100 mN load 124 ± 0.54 GPa when using a Poisson’s ratio of 0.07 and by claiming an enormous exactness (but the twinning problems were not discussed in [1]).

The known Hook values (Dl/l) are 97.2 (parallel) and 70.5 (perpendicular) GPa, the shear modulus (Hook, bending, shearing) 31.1 GPa, the bulk modulus (hydrostatic) 3.4 GPa, and the independent Young’s Moduli (Hook, resonant ultrasound spectroscopy RUS) are at 106, 87, 58, 18, 13, and 7 GPa).

These huge differences show that the most liked 124 GPa for nanoindentation of α -quartz is so far away from the various correct Young’s moduli under the varied geometric conditions, that the indentation moduli are in fact again not Young’s moduli (another confirmation of the inelasticity). It is thus clear, that the falsely derived Berkovich indentation moduli from the indentation stiffness have nothing in common. They are different in character and we are on the perfect side with indentations creating inelastic stiffness [7].

The importance of indentation moduli (these are falsely called “elastic” and they are for Berkovich falsely deduced from inelastic stiffness by using the π that is not compatible with a Berkovich indenter, but for inelastic stiffness that is the result of indentations. Nevertheless, the inelastic stiffness by indentations has more

importance than the genuine elastic ones. And the same list for applications (that was innocently formulated for elastic stiffness) can be used to them. These cover various applications that are in fact inelastic and underline their importance: that are 15 different property application-deductions of indentation stiffness. The questionable “elastic modulus” is replaced by the inelastic stiffness S [7]. We list 15 applications:

1. All elastic/inelastic properties of solids including viscoelastics
2. Elasticity Index H/S (H = hardness, preferably the physical loading k)
3. Input for FE-simulations
4. Stress-strain response σ
5. Film adhesive strength s_f
6. Adhesion calculation (DMT and JKR)
7. Creep, viscoelasticity, rheology (Kelvin Voight model)
8. Material fatigue, fatigue strength
9. Fracture toughness
10. Sliding friction
11. Contact area in continuous stiffness mode
12. Film hardness H_{eff}
13. Temperature dependence
14. Detection of phase-transitions with their onset forces
15. Search for highest reachable phase-transiting materials

This list of applications extends Table 3 in [2] (there called “elastic modulus”), which might be further extended in the future, and it spans a wide field of mechanic’s studies, but it must be physically exactly executed. The presently still misuse with elastic instead of inelastic stiffness is at high risk for daily life (one cannot “equalize elastic and inelastic stiffness and there from moduli. It is for safety reasons by developing and choosing best-suited materials for withstanding either elastic or more important inelastic stresses. They are qualified by indentation and the so found phase-transition onsets must be as high as possible, which makes the indentations indispensable. The phase-transition energies can be calculated (formulas in [3]). They must be endothermic and as large as possible.

The Value of Inelastic Stiffness

Indentations are not only important for the detection and onset of phase-transitions, but also for providing inelastic stiffness values that are totally different from Young’s elastic stiffness. The latter are determined with meticulous avoidance of any non-elastic influences. They are therefore not useful for sudden not-elastic influences upon materials, but only inelastic ones do so. These must be optimized for strong sudden impacts like car-crash, turbulence to airplanes and windmills, shoot-protection, or suddenly

overloaded bridges and buildings in earthquakes, etc. Thus, withstanding materials must have inelastic stiffness rather than only elastic stiffness. And why should we care for Young's elastic modulus if we want to avoid phase-transitions, because these are extremely dangerous for breakage and destruction that must be avoided. Clearly, local phase-transitions at the initiation site lead to interlayer between material of its transformed polymorph, and indenter diamond. In the absence an indenter such interlayer between polymorphs are building cracks and damage, which has been imaged with the test-material NaCl [6]. It was suddenly (all active airplanes within 6 week's legal checking) after appearance of [6], that the FAA (Federal Aviation Agency) understood why three airliners crashed catastrophically in short sequence, obviously due to improper material at their pickle forks (wing connections). And it grounded all the residual of this airplane series for 28 months. Their pickle fork's materials might have had a reasonable Young's elastic modulus, but that is unsuitable for sudden turbulence's attacks. The 28 months' grounding of all members of these airliner series should be long enough for the starting, and understanding the correct physics of the indentation measurements; for safer airliners without occurrence of phase-transition layers that facilitate crashing events by initial micro cracks. The sharp onset-forces of which are easily detected as kink-points in the normal force (F_N) versus $h^{3/2}$ diagram. The maximal expectable force must be considerably lower than the kink-force. If the stress exceeds the kink-force, the phase-transition will increase the breaking risk. We have here a cheap means for optimizing both factors before and after phase-transition. Why do scientists and technical Standards still not understand these facts and try with their undue "Young's elastic moduli" instead?

Conclusion

Indentations onto solid materials are unfortunately not sufficiently used for testing of technical materials at the preference of pulling, compressing, bending RUS (resonant ultrasound spectroscopy) of technical materials. These are presently preferred for obtaining elastic stiffness; the reason is certainly the false DIN-ISO-NIST-14577 Standard that unduly wants "elastic stiffness" and "elastic modulus" according to [1]. That is extremely strange; because there cannot be any doubt that the production of a stable inverted pyramidal hole is an inelastic incomplete cleavage, but not an elastic process, as outlined energetically in the preceding Chapter with exactly 20% of pressure from the applied work [6] (after the kink-point energy, for the phase-transition). Clearly, all of that is also inelastic, but the trick with load and reload tried to make people believe that they so easily obtained "elastically Young's moduli". And these are with other techniques very hard and extremely complicated to obtain. But that did produce a double drawback; a): Millions and millions of published so called "elastic stiffness" and "elastic moduli" are in fact inelastic. And they cannot tell at what force starts any phase-transition, because its onset cannot be seen with the false exponent 2 on the depth of such unphysical normal-parabola for the indentation loadings. The most important progress with such difference of elastic and inelastic stiffness or modulus is

clearly lost, and such enforcement of unphysical analyses, with the false 14577 standard, leads to worldwide catastrophes. And b): One loses the use of inelastic stiffness for characterizing and using improved materials for compliance to sudden stresses with failure due to phase-transition that could be avoided with more compliant materials, by only choosing these more compliant materials, at the expense of less compliant materials. Applications are manifold for the search and use of materials with highest inelastic stiffness, and the use is indentation, particularly nano-indentation. We can here only select some examples. The application to airliners and windmills (towards turbulences), have already been discussed above. The three catastrophic airliner crashes within two years could all have been avoided, if the author's publication [5] would not have been blocked for years by Referees (because they insisted on the false exponent "2" of [1], and the Standard 14577 on h of the loading parabola, whereas only the correct exponent 3/2 reveals the dangerous phase-transitions with their sharp onset-forces).

Some related examples are car accidents, which can be less dramatic with stiffer materials, and security cloths, and military objects against shooting impacts. Furthermore, the materials of bridges, balconies, and railway tracks etc must be optimized with highest possible inelastic stiffness against sudden overloading. The same is true for buildings against earthquakes. Clearly, chemical corrosion problems must also be avoided. And there are uncountable other events, where the inelastic stiffness of technical materials must be optimized via correct indentations for finding and selecting materials with highest inelastic stiffness.

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Conflict of Interest

The Author declares that there is no conflict of interest.

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