ISSN: 2692-5397
Modern Concepts in Material Science
DOI: 10.33552/MCMS.2024.06.000634
Iris Publishers

Mini Review

Copyright © All rights are reserved by Gerd Kaupp

A Way out of World-Wide Indentation Dichotomy in Materials' Science

Gerd Kaupp*

Department of Chemistry, University of Oldenburg, Oldenburg Germany

***Corresponding author:** Gerd Kaupp, Department of Chemistry, University of Oldenburg, Oldenburg Germany.

Received Date: June 24, 2024

Published Date: July 08, 2024

Abstract

This Short Review describes the world-wide dichotomy at the publication of mechanical materials' properties from pointed indentations, which is the most simple, rapid, and cheapest technique for obtaining these values. The dichotomy occurs when Materials' Scientists knowingly publish with "one hand" correct force versus correct $h^{3/2}$ curves and with the "other hand" ISO-ASTM-14577 enforced mechanical parameters that violate the energy-law and iterate unphysical force versus h^2 values relative values with 3 + 8 free parameters. That even though all analyses of worldwide loading curves follow exponent 3/2. They thus oppose against correct algebraic calculations. Only these reveal phase-transition onsets and mostly endothermic transition energies. These can be frozen out by indentations in liquid nitrogen where unexpectedly exothermic phase-transitions come to the fore by indentation. As negative polymorph energy content is thermodynamically poorly understood, an exciting epochal new field of research is opened.

The correct physical calculations do not violate the energy law and detect as bargain the very often found phase-transition onsets and even the transformation energies. These are of utmost importance for the optimization of technical materials. Unsuitable test materials with twinning and structural transitions transfer their errors and transitions to the fitted materials, erasing their own sharp phase-transitions. These must be known, as their onset force must never be surmounted upon technical application. It is reminded that phase-transitions form polymorph interfaces with stable micro-cracks. These are the nucleation sites for catastrophic crashes upon further increased forces. That has been microscopically imaged together with the written warning from the "new crashing mechanism". But the publication of this imaged series (including the crashing) and written warning from it was blocked by Dichotomist Reviewers and Editors for years. It could have appeared in print by Crystals, timely before the three fatal crashings of airliners in short sequence, so that all of them could have been avoided. Clearly, the Dichotomist Editor from that journal is fully responsible for all three crashings, as he additionally used quacking for the rejection. Thus, the open access publication of our very clear paper in the third of the asked journals could only appear after the fatal crashings. This appearance of the present author's publication led to a surprisingly fast reaction of FAA. It takes one six-month period for checking all operating airliners, and half a year after the appearance of my microscopic images, FAA grounded all 250 airliners for 18 months that had such (previously security-check passing) micro-cracks at their pickle-forks. FAA would have had their chance and would certainly have equally rapidly grounded, well before the three tragic crashings. The dichotomy led to these catastrophes with announcement. It reveals the enormous risk of the dichotomy. The responsibility for the three fatal airliner crashings is clear. And ISO-ASTM with the certification agencies for industries must after all follow my petitions for correcting their 14577 standard, so that the scientific dichotomy will be urgently removed for the well-being of mankind.

A face saving way to the goal of removing the worldwide shameful scientific dichotomy from both side parties is the proposed convention for an absolute instruments' standard and slogan: We need now absolute mechanical properties!

Keywords: Dichotomy; Pointed Indentations; Energy Conservation Law; Phase-Transition; Twinning; Liquid Nitrogen Effect; Crashing Prevention; Absolute Mechanical Parameters

This work is licensed under Creative Commons Attribution 4.0 License

Introduction

The necessary fight against worldwide dichotomy in materials science requires a short review in addition to a detailed publication [1]. Nanoindentation and high-force instrumental indentations are the easiest and most rapid means for the characterization of mechanical properties of solid materials. They are still worldwide performed using ISO-ASTM-14577 standardization (International Standardization Organization-American Society for Testing and Materials). Certification of involved Industries is strictly on these prescriptions that reproduce the data and claims of a 1992 paper from Oliver and Pharr [2]. This must be obeyed by them without providing them a chance for questioning the physical reality of the definitions for hardness (H as force over contact-depth square) and indentation modulus (Er as final force over initial force per contact-depth), which is almost worldwide falsely called "Young's modulus" E, after the correction for such modulus of the indenter, even though it is not an unidirectional modulus. Numerous materials' mechanical parameters are deduced from such H and E values all with unphysical results that are relative to standard plates of aluminium and fused quartz (these have to be fitted). So they drag away their errors.

The mostly applied indentations are with Berkovich or Cube Corner diamond indenters, as their geometries are well defined. But also wedged Vickers and conical indenters provide the same type of indentation law. Conversely, ISO-ASTM-14577 describes the loading curves as normal-force "FN vs h" of the basic area. Only the present German University Professor (who is legally not enforced to use unphysical formulas and data from energy-law violation) fights against such scientific exacting against him. He dared to publish, at first empirical since 1995 (by the analysis of his loading-curves' exponent and the one of published loading curves of others), and later by undeniable deducing that the relation must physically always be FN prop $h^{3/2}$ By plotting the indentation loading curves FN vs $h^{3/2}$, he obtains linear intersecting branches (sharp unsteadiness kinks) with excellent regression results also for the cases of phase-transition under mechanical load as most important bargain. Such phase-transition onset is either twinning or structural. It is extremely important and the transformation energy can be algebraically calculated. It must not be denied or handled with "work hardening iterations". Neither so is as good as possible approaching of composed loading parabolas with broken exponents (with dependence on the chosen final penetration force) physically real. The precise exponent is 3/2 (not 1.5±x) on h for uniform and, in case of phase-transitions for composed parabolas, are the physical law for pointed and wedged indentations. That has been physically and algebraically deduced, simply by consideration of the energy conservation law in 2013 [3] as the first step. The publication of the second deduction step was blocked for years by referees and editors and appeared thus later as [4]. Conversely, everybody who uses the false exponent 2 violates (and that till now) the energy law by 33.33%. He/she sincerely pretends to magically produce 33% non-penetrating work with zero energy! The correct analysis takes care of the most easily deduced fact that 20% of the applied force is used for all non-penetration work and only 80% for the penetrating work! It is so obvious for pointed indentations and wedged Vickers.

Undue Dichotomy Behavior

Due to their certification procedures all involved Industries cannot openly apt to agree with the correct physical law FN=k $h^{3/2}$ + Fa, where k is the slope of the FN vs $h^{3/2}$ plot regression, and thus the physical indentation hardness. The axis cut Fa corrects for tip rounding and all other initial effects. Dichotomists know the physical truth but nevertheless stay with the 1992 falsely claimed exponent 2 on the indentation depth h. I talked to several of these, even though that is often not making friends, because ISO-ASTM-14577 still violates the energy law and related Industries are bound to it by their necessary certification procedure and must provide the corresponding iteration procedures in their blackbox instrument computers. Academic teachers are not bound to energy-law violating ISO-ASTM standards (for a chemist these are severely exacting, and why do physicists not intervene?). But as the early publishers of indentations became the preferred anonymous Reviewers, the later Authors are either misguided or they do not dare to question the energy-law violation. Almost all of them follow blindly using the ISO-ASTM prescription, take over, and cite [2]. But the Authors of [2] were apparently only trained with iterations, but not with very basic physics and algebra. That is for hushing up the fact that experimental loading curved do, of course, not "obey" [2] and ISO-ASTM. The latter guess is particularly valid for the Authors of [5] who falsely and disdainfully called the physical law for analyzing indentation loading curves "Kaupp-fitting" instead of "Kaupp plot analysis". They rather use a quacking pseudo "deduction" of h^2 for the loading curve of pointed indentations, by putting the result (false exponent "2" from energy-law violating ISO-ASTM 14577 hardness H) in the question for obtaining it as the answer for the questioned depth exponent for the loading curve of pointed indentations.

The following Authors cite [2] and some of them even the quacking [5] for avoiding to loose friends, or with the aim to get their falsifying iterations with 3 and 8 free parameters published against their correct experimental curves, but these are simulated with "FN and h^2" or falsified by manipulation of the experimental FN vs $h^{3/2}$ data for looking as if they were "FN vs h^2" ones. That led to blocking of the present author's publications in US, Germany, and Swiss as long as possible, until the correct data were submitted to Journals with able not biased Referees. These judged with profound physical understanding and instead of rejection they encouraged for proceeding with correct physics and exact algebraic calculations.

We will see below that [5] was responsible for three fatal airliner crashes. My publication [4] was only possible, because I told the editor in my second rebuttal that the referee used details from my rejected paper for its own published paper. And I had to privately pay 3000 $ for the just started open access opening of that Journal. Clearly such problems are the roots for worldwide dichotomy that still endures (June 2024). It appears that I am still the only scientist who openly dares to comment on the energy-law violation of ISO-ASTM standards. But I and all the world profit from the bargain of the true indentation analyses with their unavoidable detection of mechanical phase-transitions, with their sharp onset points as a consequence of undeniable [3] and [4]. The closed algebraic equations for the calculations are repeatedly published, most recently in [6]. For example, the strongly denied twinning of the indentation standards aluminium and fused quartz and their structural face transitions including anisotropy has now been detected with onset-force and transition-energy [1]. That is the first reason that these are totally unqualified standards, not to speak of materials mix-up within the standards in [2] and insecure linear force application in all of the six standard materials' loading curves. My suggestion for a universal standard material is the worldwide available Zerodur[R] with two well defined phase-transitions at two well-defined rather high forces.

The indentation instrument Handbooks require to pre-scan the most unsuitable [1] (but most used) standard materials aluminium or fused quartz. Rather than analytical analyzing these (with the FN vs $h^{3/2}$ linear regression) they require their fitting to this loading curve the ones from the studied materials by two consecutive iterations with 3 and 8 free parameters, which is totally falsifying. Only a reliable standard secures the correctness of the instrument output. The materials' indentation loading curves require the linear regression analysis. The present non-physical situation can be demonstrated with the printed loading curves of the aluminium standard in the Hysitron and CSIRO-UMIS handbooks. Their FN vs $h^{3/2}$ plots give straight lines in both cases. But despite of that both insist that these reproductions be FN vs h^2 curves and both handbooks develop theory and mechanical parameter calculation on such (I am sorry) Dichotomy basis. The situation is even worse: In the CSIRO-UMIS case the linear plot of aluminium gives the correct sharp kink unsteadiness [1], as formed by two straight lines. These should at that force ranges also occur in the Hysitron case. But it is not occurring instead of only one straight line. We must conclude that their parabola is from a different material. At least it would (without revealing [1]) hide the unsuitability of aluminium as an indentation standard, because the fitting of the twinning phase-transition to the analysed materials perpetuates such face-transition unsteadiness to all of them. But that is no problem to 3 + 8 free parameters with positive or negative signs. Again, that is severe admitted Dichotomy. They get experimentally $h^{3/2}$ and feed their computer program with h^2, for instructing the people who run their instruments. A similar rather low-force twinning (their onset force is also depending on inevitable impurities at the ppm-level) occurs also with fused-quartz. For example, the false indentation hardness H-ISO-ASTM-14577 values are unphysical and violating the energy law, but how can it be prevented that multi thousands of the already published ones will become the basis of dangerous Artificial Intelligence (AI)-suggestions. These iterated H-values deny the very frequent dangerous phase-transitions! The present author analyzed hundreds of published loading curves all with such loading curves giving FN k $h^{3/2}$ + Fa regression lines and phase-transition onset force and transformation-energy. Despite of that, several authors insisted that their loading curves and thus data followed an (energy-law violating) "FN h^2" relation etc. And they missed the phase-transitions! Numerous faking examples are analyzed and very detailed rejected for different additional physical reasons in [1] and [7]. Their experimental loading curves follow always the physical formula with $h^{3/2}$. Non-experimental "loading curves" (that are simulated or faked constructed ones) are easily identified (but not by AI) and removed, but they can unfortunately not be erased. Thus AI advice will probably remain of limited value in the indentation field, unless it can be trained to recognize loading curves and calculate these with the published arithmetic formulas series, as most recently again repeated in [6]. Recognized false exponent claims data must be right-away disregarded. Most of the experimental ones were up to sufficiently high forces, so that structural phase transition onsets could be identified and recalculated. Their onsets are precisely determined (sharp kink where the two linear regression lines touch) and their normalized activation energies (mostly positive endothermic, rarely negative exothermic, mostly calculated as $\mathrm{mN\mu m/\Delta\mu m}$ units. This wealth of discovered dichotomy examples (there is no excuse for blind acting of Scientists) are collected in my open access and freely searchable online book with 265 pages and numerous figures and tables, which contains the open access indentation publications of the present author up to 2022 (including [3] and [4]) [7].

What Happens at Very Low Temperatures?

Most phase-transitions under load are endothermic and require thermal energy from the environment. But that cannot be provided at very low temperature, most easily in liquid nitrogen. So there arises the question, will be such environment perhaps suitable for exothermic phase-transitions by indentations, when all chances for endothermic ones are lost? We try to think against thermodynamic by such consideration, but why not producing thermal energy by mechanical force? We analyzed a phase-transition of an AlMg-alloy Al 7075 indentation and found from the reported loading curves one endothermic phase-transitions with a conical WC-indenter at 20°C one, two at 70°C and also two at 170°C that all disappear at very low temperature and we calculated negative (exothermic) ones that were taken at very low temperatures [8]. The analysis of the published indentation curve of Al 7075 at −196°C gave three phase transitions. The first is endotherm but with very low normalized phase-transition energy, whereas the second and third phase transitions are strongly and very strongly negative. These exothermic phase transitions lost a lot of thermal energy to the environment, which means that the produced polymorphs became negative internal energy below thermodynamic-zero. Whatever was produced, it is a new type of material and an epochal new result that cries for an advanced study by using the new in-situ onsite study possibilities with the novel Bruker cryo-in-situ tester equipment. The thermal stability of such exciting species is of highest interest. Furthermore, a rapid compression application of aluminium at very low temperature was cited in [1], but the interpretation as "new twinning path" appears too premature. But it seems promising to check it with indentation in liquid nitrogen. Important new insights will be generated and certainly achieved by indentations of further metals, salts, and oxides in liquid nitrogen or even in liquid helium. This should certainly open an as yet not thinkable new field of research and scientific comprehension.

Why is Phase-Transition Detection Important for Mankind?

Mechanical stress leads very often to local phase-transition of solids, for example with aluminium alloys for airplanes (we now interpret the TiAl issue as twinning) that needed comparatively low force for phase-transition, so that more resilient alloys towards force should be optimized for always staying below all its occurring forces. Local phase-transitions produce dangerous polymorph interfaces. I used for my paper [9] a digital microscope at 5000 fold enlargement and imaged such interfaces. The observed 1 to 1.5 µm long micro-cracks survived half a year and longer. But when I increased the indentation force, these micro-cracks grew along the interface to macroscopic cracks and finally to catastrophic crashes. I imaged also these events, termed them "a new crashing mechanism" and tried to publish at first with Scanning in USA (two very long taking rejections), then in Swiss journal Crystals, again with two long taking rejections. The second of them reasoned with [2] and with the quacking [5]. I informed the Editor in my second rebuttal on all details of the quacking (by putting the searched answer into the question) in [5], but I received immediate final rejection without any comments (a very severe Dichotomy with quacking support!).

Such publication in "Crystals" would have been the last chance to appear timely, well before the first of the three fatal airliner crashes (over China, Indian Sea, and Ethiopia). These could have been safely avoided and the stubborn Editor himself is responsible: The FAA (Federal Aviation Administration) would have reacted well before the fatal events, would it have had a chance to see my worldwide extremely important clear paper more than half a year before the first of these fatal events had occurred: The projected appearance time for "Crystals" was more than timely about one year before the first of these fatal crashes happened. But the dichotomy Editor rather favoured the quackery of [5] against my not surmountable clearly supported and written warning from crash productions due to micro-cracks from phase-transitions that are imaged in [9]. How can dichotomy so comment-less reject a worldwide helping scientific publication in favour of the quackery in [5]? Unpardonably, after the too long blocked appearance of [9], and after the actual avoidable crashes had occurred, became FAA a chance to see my micro-photographs and my new crashing sequence images in [9]. And FAA was surprisingly rapid with its extremely valuable and necessary decision. It immediately checked all airlines for micro-cracks that previously had passed these safety checks. That takes routinely 6 months for the obligatory safety check of all airlines worldwide. And FAA reacted surprisingly rapid: It grounded about half a year after the appearance of my paper all at once all of the 250 super-airliners for 18 months that had such micro-cracks at their pickle forks. That is where the wing connects with the trunk (total costs for the airliner producer and seller are 10^{11} = 100 billion Dollar). And this time period coincides closely with the time period after appearance of the open access publication [9]. Clearly, the dangerous micro-cracks in these 250 super-airliners were before present, but these were previously not complained of and passed the 6-months' safety check. Furthermore, I saw all photos of the crashing development over China in the Pilots' database and know how that developed, but I must not tell it openly.

It is the Dichotomists and in particular the rejecting Reviewers and Editors who are responsible for several years' blocking of the highly clear and understandable [9] before these 3 fatal crashes in short sequence. But now the whole world profits from my scientific fight with undeniable physics and algebra against 3 + 8 free parameter iterations as required by ISO-ASTM. My petition letter to the responsible office of ISO for compelling correction of their standard 14577 for physical reality was useless. And neither so were my well agreed research results at the yearly lectures at the respective ISO working group. It is urgent time now to again pave a way for the termination of this outrageous dichotomy in materials science.

How Can We Get Out of the Shameful Dichotomy Mess?

We must get rid of the unimaginable energy-law violation, enforcing the multiple iterations since 1992 and get rid of the producing varied relative mechanical results by fitting to very unable twinning standards. Their twinning depends on the unknowing and uncontrollable impurities content at the ppm level. The varying errors, including energy-law violation, of the standards are transformed to the studied material's results, because everything might be obtained by two iterations with three and eight free parameters! Rather we must obtain unfitted directly absolute mechanical results from checked instrumental output indentations on the basis of calculation rules that require nothing else than force and depth values with their purely arithmetic calculation using up to 6 physical law formulas.

The first task requires the checking of the instrumental outputs with a worldwide chemically uniform indentation standard that is calibrated with absolute force and depth by an able everywhere respected calibration agency for a worldwide valid absolute indentation standard. At present, the preferred absolute standard should be Zerodur^R with constant composition and precisely defined preparation for controlling or correcting every indentation instrument, so that the actual indentation result will consist of absolute force and absolute depth values. It provides two stable structural phase-transitions at high loads that can also serve as additional absolute point standards. Zerodur^R is not only worldwide used for cooking-plates, but also in heavy-duty laser optics and space-flight applications, etc. It is a well studied material.

Force and depth are all what is needed for the analyses of indentation loading curves with for example physical indentation hardness, absolute phase-transition onsets (force and depth) with their absolute transformation energies, using the repeatedly published algebraic formula series (6 simple closed algebraic most recently republished formulas in [6]).

The second task will be to get together the ISO-ASTM and Certification-Agencies party with the Dichotomists' party for the new goal, as borne from the suffered catastrophes, to stop their denying of phase-transitions under mechanical stress. After that, we hope that both parties can maintain their dignity if they claim and require with the slogan: We need now absolute results from indentations. It will only require that the instrument builders will use their possibilities to create the algorithm for the automatic calculation of the mathematically deduced formulas' sequence of the 6 arithmetic equations that are most recently re-published in [6]. After that, all archived indentation loading curves can be revived for determining the phase-transition onsets and transformation energies, notwithstanding their repetition with absolute values in cases of particular interest. And only the data determination theory part of ISO-ASTM-14577 requires revision, not their techniques for proper experimentation.

Acknowledgement

None.

Conflict of Interest

None.

References

1. Kaupp G (2024) Unexpected Twinning and Phase-Transition of the Indentation Standards, Their Transition Energies, and Scientific Dichotomy. J Appl Mathem Phys. 12(6): 2119-2159.
2. Oliver WC, Pharr G M (1992) An Improved Technique for Determining Hardness and Elastic Modulus Using Load and Displacement Sensing Indentation Experiments. J Mater Res 7: 1564-1583.
3. Kaupp G (2013) Penetration Resistance: A New Approach to the Energetics of Indentations. Scanning, 35(6): 392-401.
4. Kaupp G (2015) The Physical Foundation of FN = k $h^{3/2}$ for Conical/ Pyramidal Indentation Loading Curves. Scanning 38(2): 177-179.
5. Merle B, Maier V, Durst K (2014) Experimental and Theoretical Confirmation of the Scaling Exponent 2 in Pyramidal Load Displacement Data for Depth Sensing Indentation. Scanning 36: 526-529.
6. Kaupp G (2023) Exciting News from Indentations onto Silicon, Copper, and Tungsten. J Appl Mathem Phys 11: 4042-4078.
7. Kaupp G (2022) Basic Mathematics for Physically Correct Mechanical Properties from Indentations. Open Access e-Book from BP International. First Edition pp. 1-265.
8. Kaupp G (2023) Phase-Transitions at High, Very High, and Very Low Temperatures upon Nano-Indentations: Onset Forces and Transition Energies. Advan Mater Phys Chem 13: 101-120.
9. Kaupp G (2018) Six Polymorphs of Sodium Chloride upon Depth-Sensing Macro-indentation with Unusual Long-Range Cracks Requiring 30 N Load. J Mater Sci Engin 7: 1000473.