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CALIBRATION OF INSTRUMENTS FOR HARDNESS TESTING BY USE OF A STANDARD

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Abstract – As a standard for the calibration of force and depth scales of hardness testing instruments a system of silicon springs is described. The standard shows a linear force-deflection characteristic $F = k \cdot f$ up to a stop point (typical: F_{st} = 420 mN; f_{st} = 22 µm). For the calibration of stiffness and stop point of the standard the characteristic should be measured using a spherical indenter. In view of the practical application for the assessment of instruments also Vickers indenters can be used. In this case indentations are produced. The resulting wear can be minimized by reducing the maximum force and using only the measured stiffness *k* for a quick assessment.

Keywords: Hardness Testing, Calibration, Si-Standard

1. INTRODUCTION

Most instruments of hardness testing use electrical transducers for the measurement of force and indentation depth. Consequently, a reliable calibration and regular revisions are necessary [1], [2]. The generally used method is based on calibrated hardness testing blocks which are wearing out. For the range of hardness of viscous materials this method cannot be applied because of temporal instability.

As an alternative a silicon standard was developed produced by microtechnologies, Fig. 1. This standard is based on a linear-elastic spring system with an integrated stop point. It represents a material measure for force and dimension. The design is focused on the calibration of instruments for hardness testing. Silicon is an excellent material for calibration springs because of its elastic behaviour which is free of effects of hysteresis and fatigue. Additionally, silicon has a minimal thermal expansion and a remarkable hardness. Finally, the processes of silicon microtechnique guarantee the production with precise dimensions and stable parameters.

2. DESIGN AND MANUFACTURE OF THE STANDARD

The standard should fulfil the following requirements:

- Linearity: constant stiffness $k = F/f$ < 40 mN/µm
- Detection of a final deflection of max. f_{st} = 25 µm at a definite force F_{st} < 1 N by contacting a stop point (abrupt increase of stiffness)
- Compliance of the stop point to avoid wearing (limited increase of stiffness from k up to $k_{st} > k$)
- Sharp transition of the characteristic $k \rightarrow k_{st}$ at f_{st}, F_{st}
- Insensitivity of these parameters to small variations of the contact position.

2.1. Design

A spring system best fulfils the last point if it has a symmetrical shape. In this case the dependence of the deflection on position of loading shows an extreme in the centre and varies here only minimally.

An exact linear characteristic can be reached using a system of parallel springs like a vertical straight guide. A central boss plate is useful for loading by an indenter. At the corners the boss is fixed by leaf springs which are connected at their other ends with two joining bars. At the ends these joinings are fixed by leaf springs to the frame. Fig. 2 shows the used symmetrical variant. The force F necessary for a deflection f can be estimated by the analytical relation (1) (E: Young modulus of the length direction of the springs; b, h, L: width, height, length of the springs, k: stiffness) [4].

$$F = \frac{2Ebh^3}{L^3} f = k \cdot f \qquad (1)$$

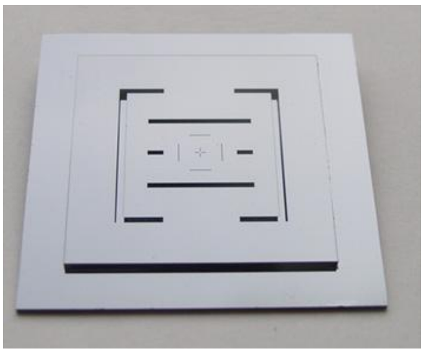


Fig. 1. View of the calibration standard. In the centre the marks for position of loading can be seen. The support-chip is larger than the spring-chips for a better handling.

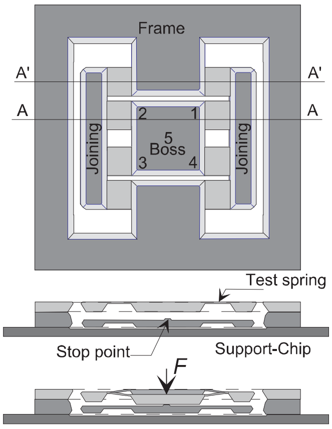


Fig. 2. Scheme of the standard. Top: backside of the chip with the test spring. Bottom: cross sections A-A and A'-A' of the system without and with loading respectively

Increasing load produces a linear deflection of the boss up to the contact with a stop point realized as a hillock on a second spring system lying underneath. Reaching this stop point the stiffness will be increased drastically indicating the deflection f_{st}. Consequently the standard consists of three chips: the test spring, the spring with the stop point and a support-chip for better handling, Fig. 2 bottom.

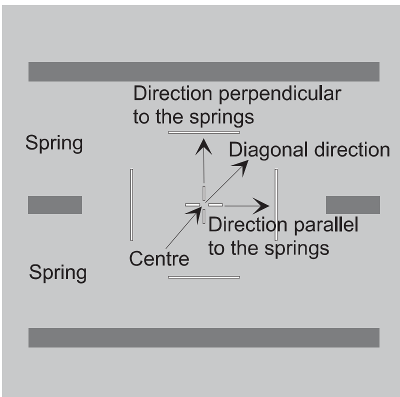


Fig. 3. Scheme of the eccentric load positions.
Range inside the cross: 300 µm x 300 µm.

The layout of the spring system was orientated parallelly or perpendicularly to the flat of the {100}-silicon wafer. So the Young's modulus E corresponds with its value along the <110>-direction ($E = 169$ GPa). Considering the small variation of the elastic moduli with the temperature ($\Delta E/E \approx -1 \cdot 10^{-4}$ K^{-1}) and the small thermal expansion of silicon ($\alpha = 2.6 \cdot 10^{-6}$ K^{-1}) [3] a small relative change of the stiffness $\Delta k/k$ can be estimated in the order of 0.02 %/K.

The target values of the spring system are achieved by the design of its dimensions described in more detail in [4]. The outer dimensions of the standard are 40 mm x 40 mm with the height of 1.6 mm. The finding of the central region for loading is supported by etched grooves, Fig. 3.

2.1. Manufacture

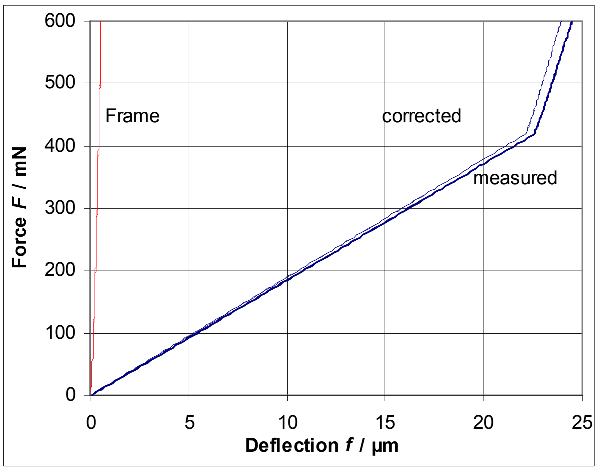
The standard is manufactured out of silicon wafers by microtechnological processes. Each silicon wafer contains four identical chips. For 3D structuring of the three types of chips the KOH-etching is used. The wafers with the chips of the test springs and the wafers with the stop point springs are mounted together by silicon fusion bonding. The resulting stack is silicon fusion bonded or glued by a hard wax on the wafer with the support chips. Dicing the mounted wafer stacks yields up to four standards. These processes are described in more detail in [4].

3. RESULTS AND EXPERIENCES

The produced standards were tested in five laboratories with different instruments. The tests should expose the parameters of the standard in view of its linearity, the values of stiffness and stop point deflection and the repeatable accuracy of these parameters. Further, experiences of use of the standard should be gained as the realizable precision of loading point, the stability of positioning and the influence of indentations. From these experiences a guidance for the use of the standard for the assessment of instruments can be derived.

3.1. The parameters of the standard

The parameters of the standard were determined by loading with a spherical tip to avoid indentations.



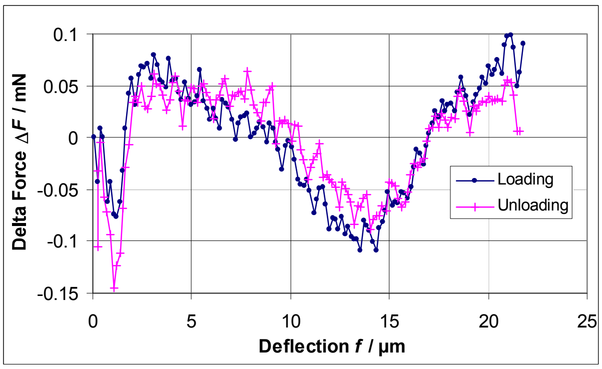


Fig. 4. Top: Characteristics $F(f)$ measured on the standard and the frame and the corrected characteristic of the standard
Bottom: Deviations from the linear fits of the corrected values (standard 5-2; instrument 4; diam. of sphere: 50 µm)

Linearity

As expected the standards show an excellent linear behaviour over the full region of deflection up to the stop point. No hysteresis occurs, Fig. 4.

Some conditions must be kept or considered:

- the standard must have a stable position on a rigid support with a flat surface free of dust
- no plastic or viscous deformations are allowed anywhere in the measuring system
- the elastic deformation of the measuring system (instrument, indenter, silicon surface) must be eliminated.

The last effects can be integrally captured by measuring a load-deflection characteristic on the stiff frame of the standard and subtracting it from the characteristic measured on the test spring. Fig. 4 shows the result. The plots contain the loading and unloading characteristics.

The corrected characteristic of the test spring can be fitted by the equation

$$F = k \cdot f + F_0 \qquad (2)$$

As an example Table I contains the values of k and F_0 together with their standard deviations Δk, ΔF_0 and the deviation from zero $f(F_0)$ resulting from the characteristics shown in Fig. 4.

TABLE I. Stiffness and parameters characterizing the linearity

Loading			Unloading		
mN/µm	mN	µm	mN/µm	mN	µm
k 18.934	F_0 0.008	$f(F=0)$ 0.000	k 18.936	F_0 0.100	$f(F=0)$ -0.005
Δk 0.001	ΔF_0 0.010		Δk 0.001	ΔF_0 0.008	

The values of the force calculated using (2) differ from the corrected values no more than ± 0.1 mN, Fig 4 bottom, indicating the good linearity of the standard.

Deflection f_{st} at the stop point

If the boss is deflected up to the hillock on the spring underneath the stiffness will be increased. A kink in the characteristic is produced as can be seen in Fig. 4.

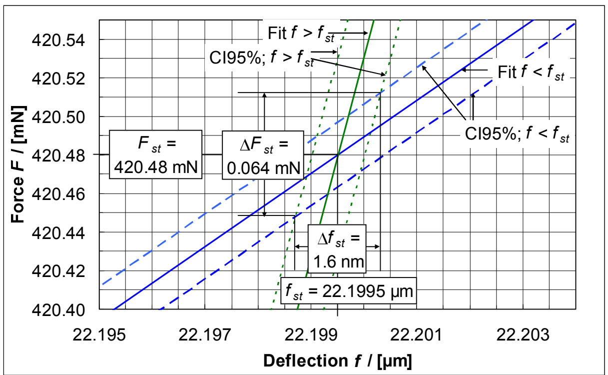


Fig. 5. Determination of the stop point deflection f_{st}. (standard 5-2; instrument 4)

Because of the sharpness of the kink the quantity f_{st} can be exactly determined and used as a material measure for the assessment of the scale of length of the instrument. Fig. 5 illustrates this detail of the characteristic shown in Fig. 4. The slope is changing from one point to the next resulting in the distance of these points as uncertainty of f_{st}. Calculating the point of intersection of the linear fits before and after the stop point the value of f_{st} can be determined with more accuracy. Considering a confidence interval (CI) of 95% the uncertainties of the point of intersection ΔF_{st}, Δf_{st} amounts to 0.064 mN and 0.0016 µm respectively.

Influence of eccentric load positions

To assess the sensitivity of the standard to eccentric loading a series of characteristics was measured by variation of load position d along the directions indicated in Fig. 3.

It can be seen that the falsification of the stiffness remains below 3 ‰ if the load position is in the 300 µm × 300 µm square inside the cross bars.

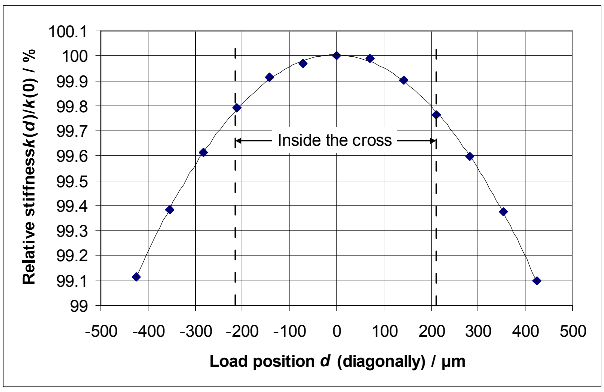


Fig. 6. Variation of the stiffness under eccentric load. (standard 5-2; instrument 4)

3.2. The practical application of the standard

In practice the standard should permit an assessment of a hardness testing instrument, that is the verification of the scales of force and indentation depth. Usual methods are the verification of the force by an electronic balance and of the depth by an interferometer. These procedures need a considerable expenditure of instrumentation and time. In view of a quick assessment the standard can be advantageously used. Three different procedures are realizable presupposing the standard itself is calibrated.

i *The instrument is equipped with a spherical indenter with a large radius.*

In this case the procedure is similar to the measurements described in 3.1 resulting in values of stiffness k and deflection f_{st}. Unlike as described in 3.1 the instrument must be adjusted up to the certified values of the standard.

ii *The instrument is equipped with a* Vickers (or Berkovich) *indenter – measuring the full characteristic.*

If the standard is used in an instrument with a Vickers (or Berkovich) indenter and if the characteristic is measured up to the stop point an indentation will be produced together with cracks and particles are broken out, Fig. 9. The loading branch of the characteristic reflects the plastic deformation and the creation of cracks resulting in nonlinearity and a depth difference to the branch of unloading (see Fig. 7). If the measurement is repeated without changing the position, the branches of the characteristic are nearly identical.

The branches of loading and unloading contain further the elastic deformation of the instrument itself (instrument compliance) and of the silicon surface. Analogous to section 3.1 these deformations can be measured by an indentation experiment on the frame of the standard. Subtracting the load-deflection curve of the indentation on the frame from the curve obtained inside the standard, the values of stiffness and stop point deflection can be calculated, Fig. 8.

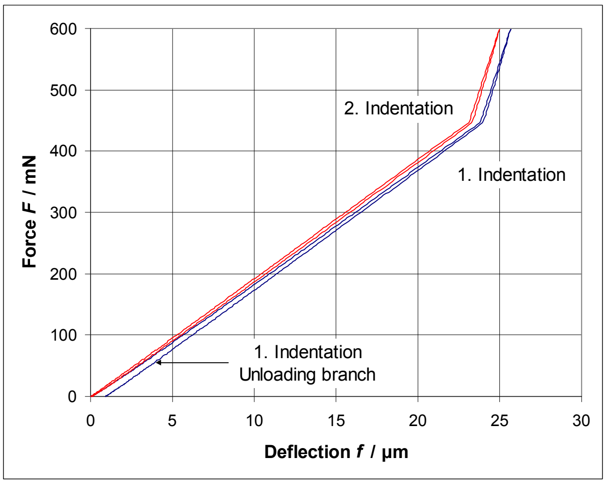


Fig. 7. Characteristics of the standard measured with Vickers indenter. (standard 8-2; instrument 5)

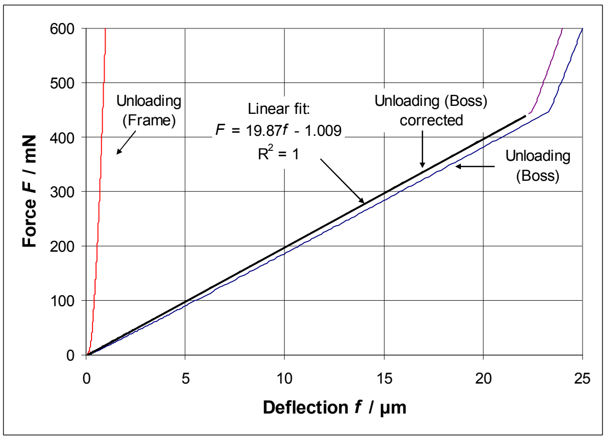


Fig. 8. Corrected unloading branch of the standard. (Vickers indenter, standard 8-2; instrument 5)

The slope of the corrected unloading branch results in the measured value of the stiffness. The values F_{st} and f_{st} at the stop point can be determined from the corrected unloading branch as described in section 3.1, Fig. 5. Then the instrument must be adjusted up to the correspondence of these values to the certified values of the standard.

The comparison of the parameters measured with a spherical or a Vickers indenter shows small deviations and more unique results for the sphere, Table II. Consequently, the spherical indenter should be used for an accurate calibration.

TABLE II. Comparison of the characteristical parameters of the standard measured with different shapes of indenter for the 1st measurement and repeated measurements.
(standard 8-2; instrument 5; diam. of sphere: 2 mm)

Indenter	k mN/µm 1st meas.	k mN/µm rep. meas.	f_{st} µm 1st meas.	f_{st} µm rep. meas.
Sphere				
loading	20.408	20.373	21.903	21.948
unloading	20.386	20.388	21.900	21.950
Vickers				
loading	20.028	19.897	22.308	22.424
unloading	19.964	19.799	22.374	22.515

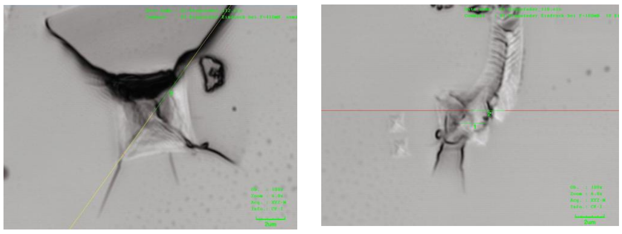


Fig. 9. Characteristic Vickers indentations on the boss. Left: F = 410 mN Right: 10 indentations F = 100 mN (standard 6-2; instrument 3)

iii *The instrument is equipped with a* Vickers (or Berkovich) *indenter – partial measuring of the characteristic.*

Because of the production of indentations on the boss plate the surface region inside the cross will be worn out. Using an indenter of Vickers or Berkovich type wearing can be only avoided by limiting the force to the elastic region. This limit depends on the tip radius r of the indenter and amounts in silicon to less than 50 µN at $r \approx 0.2$ µm.

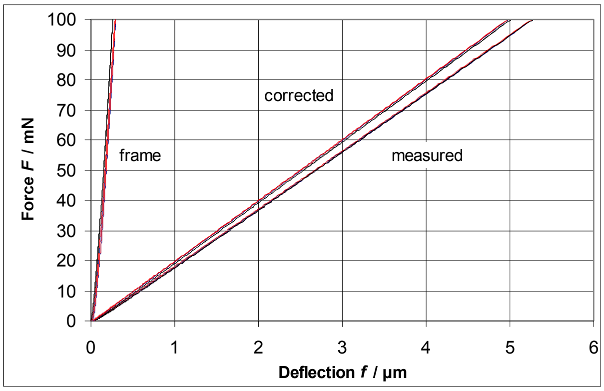


Fig. 10. Three characteristics (slightly differing positions) measured up to F_{max} = 100 mN and the corrected characteristics (Vickers indenter, standard 8-2; instrument 5)

In the case of forces in this order of magnitude only the stiffness of the standard can be used for a quick assessment of the instrument. Loading up to a force of F = 100 mN smaller indentations are produced and, consequently, less wearing, Fig. 9 right. The resulting characteristics measured as the 2nd or higher repetition indicate no plastic deformation. After correction by subtraction of the deformation of the measuring system (2nd or higher repetition of measurements on the frame) linear and nearly identical branches of loading and unloading result, Fig. 10. The stiffnesses k are close to the values determined for a spherical indenter as shown in Table III.

TABLE III. Stiffnesses determined from characteristics up to 100 mN corresponding to Fig. 10

	k mN/µm No. 1	k mN/µm No. 2	k mN/µm No. 3
loading	20.179	20.193	20.148
unloading	20.167	20.202	20.178

3.3. Experiences in practical use of the standard

Using the standard with different instruments some practical experiences were made which can be divided into two groups related to a certified calibration of the standard and to its use for calibration or assessment of instruments.

In both cases the demand for a stable positioning of the standard is indispensable. An uneven support can result in unwanted bending or tilting effects. In the measured deflection an unknown contribution results, which can be different for loading the boss or the frame, so that the correction of the stiffness of the measuring system fails. This problem can be solved by glueing the standard with wax on an evenly ground steel plate.

Loading with a spherical indenter produces very unique and reproducible results suitable for a certified calibration of the standard. Indenters of Vickers or Berkovich type produce indentations during the 1st loading. Unloading and repeated loading without changing the position produce no additional plastic deformation (the loading branches of repeatedly measured characteristics are nearly identical with the unloading branches). In practice differences can occur if asymmetries of the instrument and a mobility of the standard change the position. A series of indentations is produced, Fig. 9 right, influencing the measured characteristics. The disappearance of this effect can be expected if standards mounted on a steel plate are used.

4. CONCLUSIONS

The described silicon spring system is a material measure for forces and depths of about 0.4 N and 22 µm respectively. It can be used as a standard for the assessment of hardness testing instruments. The characteristic parameters F_{st}, f_{st} and the stiffness k of a standard determined under reliable conditions allow the certification. The application for calibration of a hardness testing instrument equipped with a Vickers (or Berkovich) indenter produces plastic deformation (indentations) during the loading process. Consequently, for the verification of the instrument repeated indentation measurements should be the base for the determination of stiffness. In addition the elastic deformation of the measuring system (the instrument, the indenter and the silicon surface) has to be eliminated by subtraction of load-deflection curves measured on the frame of the standard.

REFERENCES

[1] DIN EN ISO 14577: Instrumented indentation test for hardness and other materials parameters - Part 1: Test method - Part 2: Verification and calibration of testing machines.

[2] Ch. Ullner, A. Wehrstedt, „Determination of Martens hardness, indentation hardness or indentation modulus - Instrumented indentation test for hardness after ISO/DIS 14577“
„Martenshärte, Eindringhärte oder Eindringmodul ermitteln - Instrumentierte Eindringprüfung nach ISO/DIS 14577“, *Härtereitechnische Mitteilungen* 56. Jg. (2001) 4, S. 241-248.

[3] EMIS Datareviews Series No. 4: “Properties of Silicon”, INSPEC, The Institution of Electrical Engineers, Lomdon New York 1988.

[4] J. Frühauf, E. Gärtner, K. Herrmann, F. Menelao, „Standard for calibration of instruments of hardness testing“ „Kalibriernormal für Geräte der registrierenden Härteprü-fung“. *TM Technisches Messen*, in press.

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