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VERIFICATION OF THE STANDARD MACHINE FOR THE MACRO RANGE OF INSTRUMENTED INDENTATION TEST

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Abstract – A new calibration machine according to the requirements given by ISO 14577-3 [1] was described in [2]. The results of the verification are reported in this paper. It is shown that the calibration machine can be used as a national standard for the materials parameter of the instrumented indentation test.

Keywords: indentation, standard, verification

1. INTRODUCTION

Although the standard ISO 14577 on the instrumented indentation test was issued 2002 [1], the metrological traceability has not been completed in the macro range up to now. A new calibration machine according to the requirements given by ISO 14577-3 was described in [2]. The high precision of the generated indentation curves including force and displacement measurement as well as the machine compliance should be proved in this paper.

2. MACHINE DESIGN

The machine should be used up to 1500 N with the ability to reach the upper micro range according to the definition in ISO 14577 [1]. Force measurement down to 1 N should be done with a measurement uncertainty of 5 mN. Displacement measurement of the indenter should be accomplished with uncertainties of 9 nm over a 220 µm displacement.

The capability for the calibration of reference specimen is limited by the uncertainty in compliance of the machine. The aim is to achieve an uncertainty in determining the compliance of 0.5 nm/N on those parts of the machine which have a direct effect on displacement measurement.

The machine design principles are described in detail in [2]. The foremost design principle – to achieve an optimum behaviour of the compliance affecting the displacement measurement – is given by an inverted layout. Fig. 1 shows the principal layout of the machine with the specimen adjacent to the thrust bearing on top of the separated indenter-support. Force transducer and indenter-support are axial restraint by a leaf spring system, which guides the movement created by the piezotranslator. In conjunction with the inverted layout and the fixed thrust bearing, the position of the indenter-tip is connected to the individual specimen thickness. The satellite roller screw is able to travel the indenter the required ways and places the unit piezotranslator / force transducer / indenter in the proximity of the specimen while clamping the specimen simultaneously.

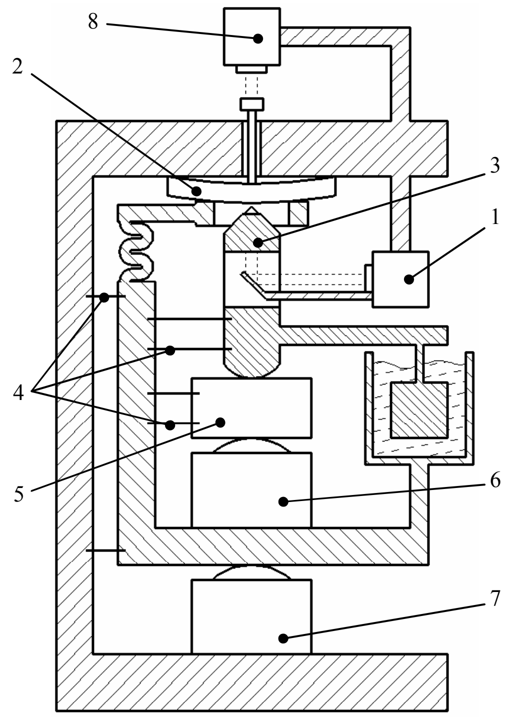

Fig. 1 Schematic layout of main systems: 1 first laser interferometer with support bar and tilted mirror, 2 specimen, 3 indenter with support frame and reflective mirror, 4 leaf spring linear guidance, 5 force transducer with precision mount, 6 piezotranslator, 7 satellite roller screw with servo drive , 8 second laser interferometer with tracer pin

3. FORCE CALIBRATION

Force calibration of the machine is possible through a tracer pin, which is normally used to determine changes in the position of the back of the specimen in order to detect possible bending or movements in the contact area to the thrust bearing. With the specimen removed, the tracer pin permits calibration or plausibility checks of the built-in force transducer by placing calibration weights on the tracer pin (Fig. 2).

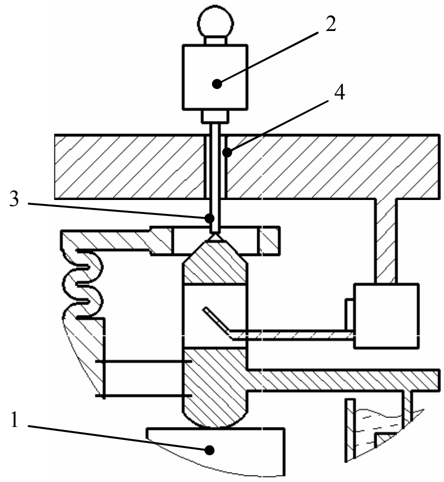

Fig. 2. Calibration of the force transducer (1), using a calibration weight (2) in connection with a tracer pin (3) by utilizing the drill hole (4) in the thrust bearing

Such a direct check as given by ISO 14577 part 2 is conducted either with a set of calibration weights or with a force-standard according to ISO 376. Care must be taken for using additional elements such as the tracer pin, because they may add further uncertainties through their guidance elements.

The wide range of applied force requires the use of exchangeable force transducers. To avoid an additional source of uncertainty in force measurement a precise mount was designed, where the force transducer is held in place but can be easily exchanged. The exchangeable component of the mount is permanently attached to the force transducer.

A comparison of the two types of force calibrations (transducer outside the machine using the standard calibration machine and transducer inside the machine including several removals) has shown that there is no need for further calibration after exchanging the transducer. The new designed mount permits calibrating the force transducer outside the calibration machine in the optimal way according to ISO 376 using the standard calibration machine (Tab. 1).

Table 1. Comparison of the calibrations of the 200 N force transducer (outside the machine using the standard calibration machine and inside the machine including several removals)

	20 N	40 N	60 N
Mean outside (N)	20.00064	40.00128	60.00159
Mean inside (N)	20.00109	40.00218	60.00327
Standard deviation outside (N)	$<10^{-7}$	$<10^{-7}$	0.00047
Standard deviation inside (N)	0.00029	0.00058	0.00088

4. LEAF-SPRING CORRECTION

The ability to exchange force transducers requires the need for a separated indenter-support. The indenter is not directly attached to the force transducer and must be replaceable and separated from the force transducer, but should be guided in such a way, that only linear movements in the axis of the indenter are possible to fulfil Abbe's law.

Precise axial guiding of the indenter support is accomplished by utilising a leaf spring linear bearing as described in [2]. As can be seen in Fig. 1, the piezotranslator moves the force transducer and the indenter in the direction of the specimen. Although the displacement of the indenter is only 300 µm at maximum – minus the contraction of the force transducer and piezotranslator through compression – the 16 single leaf springs of the linear bearing may have a significant contribution to the uncertainty of force measurement.

Using very thin leafs as the first approach failed to achieve a working linear bearing. With changes well below 5 mN over a 300 µm displacement, it had not seriously affected the desired uncertainty in the lower force regime down to 1 N, but resistance against side forces and proper linear movement were not given. Using significantly thicker leaf springs lead to a precise working linear bearing with force changes in the region of 200 mN over the maximum range of displacement.

To check the effect of the spring system, the additional force was measured during the travel of the indenter-support frame (Fig. 1). The detailed force dependence is given in the following polynomial

$$\Delta F = -0.0002x^2 + 0.66x \quad , \qquad (1)$$

ΔF is the additional force in mN and x is the displacement of the spring system in µm. The spring constant

$$C_s = \frac{d\Delta F}{dx} = -0.0004x + 0.66 \qquad (2)$$

is poorly dependent on the displacement of the piezo-translator. Its range is $0.54 \le C_s \le 0.66$ mN/µm at movements from 0 to 300 µm. Assuming uniformly distributed displacement, the uncertainty of the spring constant is [3]

$$u(C_s) = \frac{0.06}{\sqrt{3}}\frac{mN}{\mu m} = 0.035\frac{mN}{\mu m} \quad . \qquad (3)$$

Using the Martens hardness HM [1] and the indentation depth as the spring displacement, the relative uncertainty of force due to the corrected spring-effect reads

$$w(F_{C_s}) = \frac{u(C_s)}{\sqrt{26.43 HM \cdot F}} \quad . \qquad (4)$$

With the minimum HM = 500 N/mm^2, it can be concluded from equations (3) and (4) that the estimated relative uncertainty is less than 0.1% at the test load of 0.1 N. At test loads above 33 N the estimated relative uncertainty is less than 0.1% due to the small spring constant of C_s = 0.66 mN/µm even if the correction (1) has been omitted.

Prior to a measurement campaign the spring constant C_s should be determined. The piezotranslator should be used to record a graph of the spring-effect on force measurement with unloaded indenter by going through the full range of displacement.

5. DISPLACEMENT CALIBRATION

The ability to calibrate force and displacement with built in sensors, as given by ISO 14577, is feasible. Because of the low measurement uncertainty of the used laser interferometers, a better sensor is hardly available. However, a comparison of the measured values between both interferometers appears meaningful (Fig. 3).

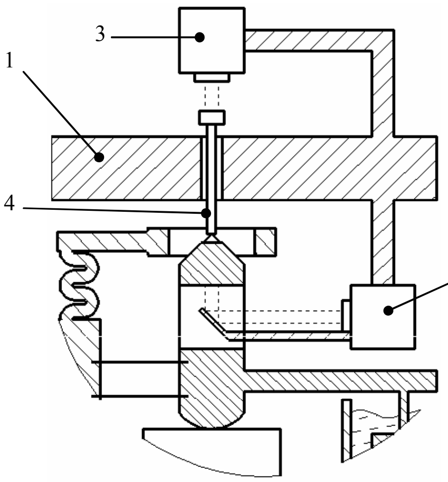

Fig. 3. Thrust bearing (1) with drill hole. A comparison of measured values of the primary (2) and second laser interferometer (3) in combination with a tracer pin (4) is possible

6. MACHINE COMPLIANCE

As stated earlier [4], the uncertainty in compliance on those parts which have a direct impact on displacement measurement is responsible for the ability to calibrate reference specimen. Through careful design, the engineer is able to delete or weaken most of the undesired sources of compliance.

The inverse layout (Fig. 1) establishes stable conditions by using gravity to preload the parts and joints in such a way, that load alternation or a zero force load state is eliminated. All parts, including the outer frame and sensor chain, are preloaded by gravity in the same direction as the direction of the additional load during the measurement process.

The two laserinterferometers for displacement measurement are attached to the thrust bearing in such a way, that possible bending of the bearing in consequence of force influence does have the same magnitude of influence on both sensors. In theory, bending of the thrust bearing does not contribute to the uncertainty in displacement measurement at all. Because of the arrangement of the primary laserinterferometer and the tracer pin with the second interferometer in the line of the indenter, all movements of the specimen during measurement are detected and used to correct the signal of the primary laserinterferometer.

Furthermore, indenter compliance is minimized by clamping the indenter at the outer flange with a ring to reduce contact movements with the indenter support during indentation process. The important indenter mirror is axially guided by a small leaf-spring linear bearing and spring loaded onto the indenter-shaft to minimize the effect of possible deformation of the support frame due to the applied force during measurement (Fig. 4).

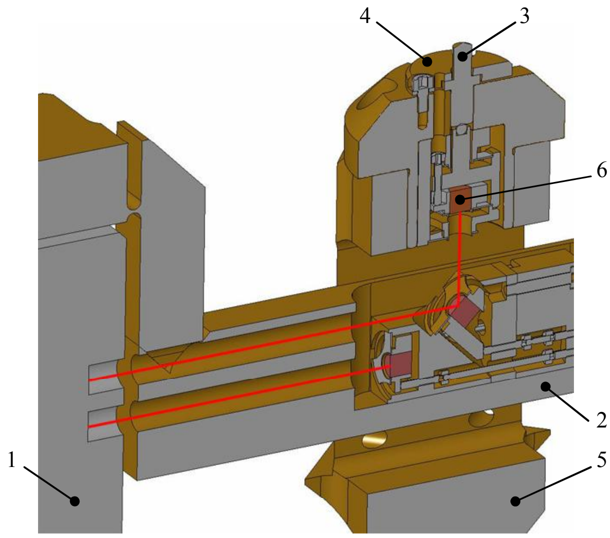

Fig. 4. Indenter support with displacement sensor: fixed laser interferometer (1) with support bar and tilted mirror (2), indenter (3), indenter clamping ring (4), indenter support frame (5), axially guided reflective mirror (6)

To determine the machine compliance, 11 tests have been conducted using a Vickers indenter and a hardness reference block 540HV30. For application and removal of the test force, the selected displacement rate of the piezotranslator was 2 µm/s. The time up to the maximal force of 200 N and the holding time were 110 s and 10 s, respectively.

Assuming the hardness is independent of the force in the macro range, the following polynomial of 2^{nd} order for $h(F^{0.5})$ is valid for the force application [4]

$$h_{measured} = h_0 + \sqrt{\frac{F}{G \cdot HM_S}} + C_m \cdot F \, , \qquad (5)$$

with G – geometrical factor, HM_s – Martens hardness determined from the slope [1], and C_m – machine compliance. All results of the 11 tests are plotted in Fig. 5. Except for the range of small indentation depth the precision is excellent. To avoid the effect of the rounded indenter tip, the curves in Fig. 6 are fitted within the range between 6 µm and 200 N. The mean values and the standard deviations of the 22 parameters in (5) are

$$\sqrt{\frac{1}{G \cdot HM_S}} = (2.773 \pm 0.003)\mu m N^{-0.5} \qquad (6)$$

$$C_m = (0.00477 \pm 0.00019)\mu m N^{-0.5} \, . \qquad (7)$$

The mean value of C_m show that the undesired machine compliance is limited to the loaded indenter between the tip and the flange only.

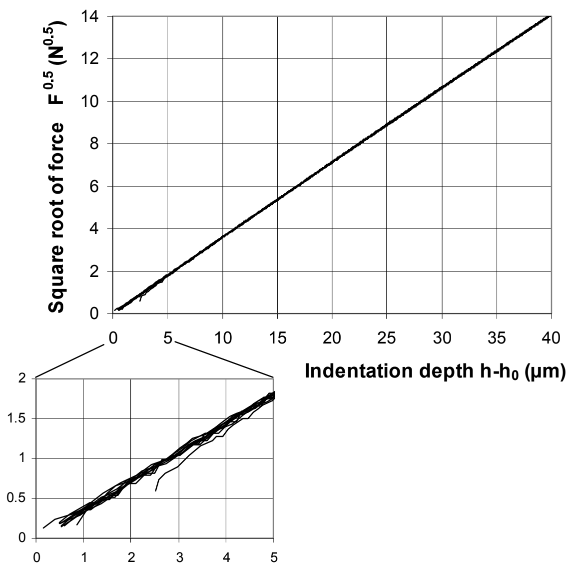

Fig. 5. All results of the 11 tests plotted by using the fitting parameter h_0 of (5)

The excellent precision of the machine compliance is impressive. Although an extended estimation of uncertainty has to take into account additional sources [5], the results show that the design of the machine allows limiting the uncertainty of the machine compliance to 0.5 nm/N.

In addition, it should be checked if the machine compliance is independent of the test force. For this reason, a specified plot of the indentation curve is chosen in Fig. 6 [4]. The resulting curve is extremely sensitive to any scatter of the measurement. The independence of the machine compliance is proved for a test force greater than 7 N, because the slope in Fig. 6 is formed by the machine compliance.

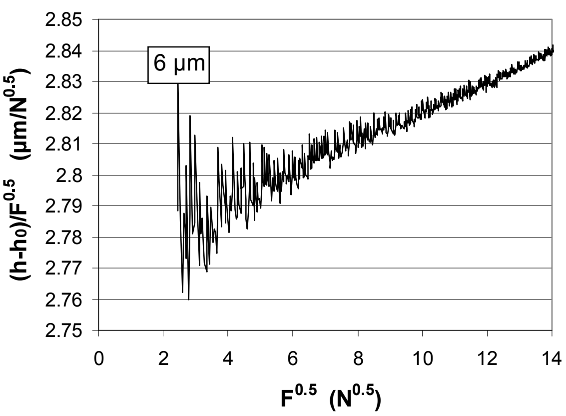

Fig. 6. Demonstration of the check, if the machine compliance C_m is independent of the test force. The slope of the curve is equivalent to the machine compliance

7. DISCUSSION

While the small uncertainty of the machine compliance is the most important assumption for a standard machine in the macro range of the instrumented indentation test, the very small standard deviation of the determined parameter in (5) and (6) is also an improved result related to the results of the commercial machines. The Martens hardness HM_s, determined from the slope [1] can be given with the relative expanded uncertainty (k=2) of 2% [5]. Because of the fluctuations of the displacement in the range near the contact (Fig. 5), the scatter of the maximum indentation depth h_{max} (including the Martens hardness HM and the indentation hardness H_{IT} [1] as a consequence) is little greater than the scatter of HMs. It should be noted that the test force at a indentation depth smaller than 6 µm is dependent on the specified real geometry of the indenter and the surface roughness [1], i.e. only the standard value of HM_s (and not HM) is independent of the indenter tip. Therefore, it should be discussed if the Martens hardness HM, and the indentation hardness H_{IT}, are appropriate for the metrological chain to prove the traceability.

The very high precision obtained in the determination of the machine compliance can be achieved by following Abbe's law. It is worth mentioning that the displacement measurement with the second interferometer at the back of the specimen (Fig. 1) has shown a displacement up to 2 µm under the test force of 200 N. Such a displacement occurs although the tested hardness reference block is 6 mm thick with a very even surface. However, if such a displacement is caused by tilting or bending then incorrect displacement is measured by every machine which does not fulfil Abbe's law. For instance, machines for the macro range of the instrumented indentation test have a displacement measurement related to the surface which the indenter is driven into. That means, the reference point is located out of the force axis about 3.5 mm at minimum (for a standard indenter). Assuming a specimen with a length of 70 mm is tilting by 2 µm, the reference point is being shifted by about 200 nm related to the indenter tip. In this way, the compliance of the conventional machine is measured incorrectly by about 1 nm/N. Taking into account that tilting or bending of the specimen do not give a linear interrelation between force and displacement, the additional virtual compliance can overlay strong fluctuations over the indentation curve up to the maximum force.

Fig. 7 gives an example of the precise shape of the indentation curve. The high precision of the indentation curve can be obviously seen in the range where the indenter is removed from the specimen. The ability to guide the indenter separately from the force transducer minimises the impact of side forces during indentation and seems to reduce the measurement uncertainty. There is a sharp transition from the force removal to the force free state.

In terms of correcting the effects of the leaf spring linear guidance on force measurement we should note that the return slope after separating the indenter from the surface of the specimen can be used to correct the spring-effect.

The high precision of the test results allows checking the homogeneity of the chosen reference material. In our experience, the available reference blocks for the conventional hardness tests are also well appropriate for the instrumented indentation test [6]. However, the new machine gives the chance to check the homogeneity of further materials more sensitive.

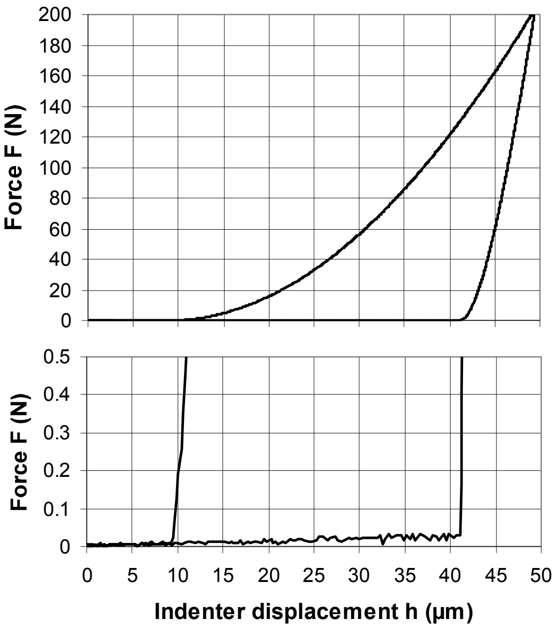

Fig. 7. Demonstration of the precise shape of the indentation curve including the zoomed range from the force removal to the force free state (below)

8. CONCLUSION

The first validation of the machine has shown that the national standard for the macro range of instrumented indentation test can be provided. A discussion on a key comparison in the macro range should be initiated as the next step. All efforts in minimizing measurement uncertainty and avoiding zero force states incorporated in the design proved effective. In fact, tests in the upper micro range may also be possible in future.

In addition, the mechanical design has the potential to assist in further development of the standard. Using innovative design features, the new machine design may lead to a standard solution not only for calibration machines.

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