Mechanical Characterization of Material Coatings Using the Fischerscope® H100 Compakt

Dipl.-Ing. P. Neumaier (Helmut Fischer GmbH +Co. KG – Sindelfingen)
and Dr.-Ing. habil. G. Michalzik (Ulm)

1.Introduction

The load/indentation depth method has received broad acceptance both in quality control and in research and product development for characterizing the mechanical behavior of thin coatings and also of small and smallest material areas. Continuous high-resolution recording of test load and indentation depth for the full test cycle (loading and unloading) is used in a variety of ways in particular for test loads in the microhardness range. Contributing factors to its acceptance were the greater information content of the measurement results, the operator-independent test procedure and the speedy standardization of the test method.
However, until now, the use of the instrumented indentation test has been limited to relatively small specimens or required the supply of small samples. Extracting a sample leads to the destruction of the product, and the separate production of a sample requires additional expenditures and does not always ensure comparable properties.
The computer-controlled Fischerscope® H100 Compakt (H100 C) opens entirely new areas of applications for efficient tests of materials and thin coatings both on small samples/micro components and on large specimens such as coated shafts, forming components, etc.
Using selected examples, this paper reports about the capabilities of this new measurement technology for applications with small test loads and indentation depths as well as the use of the mobile measuring head H100SMC on large-area and compact specimens.

2. The new measurement and test technology

The core of the Fischerscope® H100 C is the compact measuring head H100SMC (Figure1) that can be expanded to a test system with versatile applications / 1-3 /.
A replaceable indenter, the load generation and the distance measurement systems as well as the entire electronics are integrated in the measuring head. The control and evaluation unit consists of a commercial PC with the WIN-HCU® Software installed. This software provides all functions for controlling the measuring head electronics, setting the test parameters as well as for the convenient evaluation, presentation, documentation and archiving of the measurement data and the characteristic data derived from them. When connected to a positioning device, it also assumes its control.
The operator also has the ability to define the test cycle – maximum test load, load and unload times, hold times at maximum load or at zero load after unloading – as well as all standardized characteristic data according to the test application. Additional measurement sequences such as cyclic loading and unloading with the same or different end loads can be implemented using Microsoft® EXCEL.
In its most simple application, the measuring head is manually placed on the test area selected by the operator. The test cycle runs automatically load-controlled after starting it with the PC key.

The new measurement technology features the following instrument parameters:

Test load range: 0.4 to 1000 mN **Load resolution**: 0.2 µN
Distance measurement range: 400 µm **Distance resolution**: 0.1 nm
Approach speed of the indenter towards the test area: 2 µm/ s

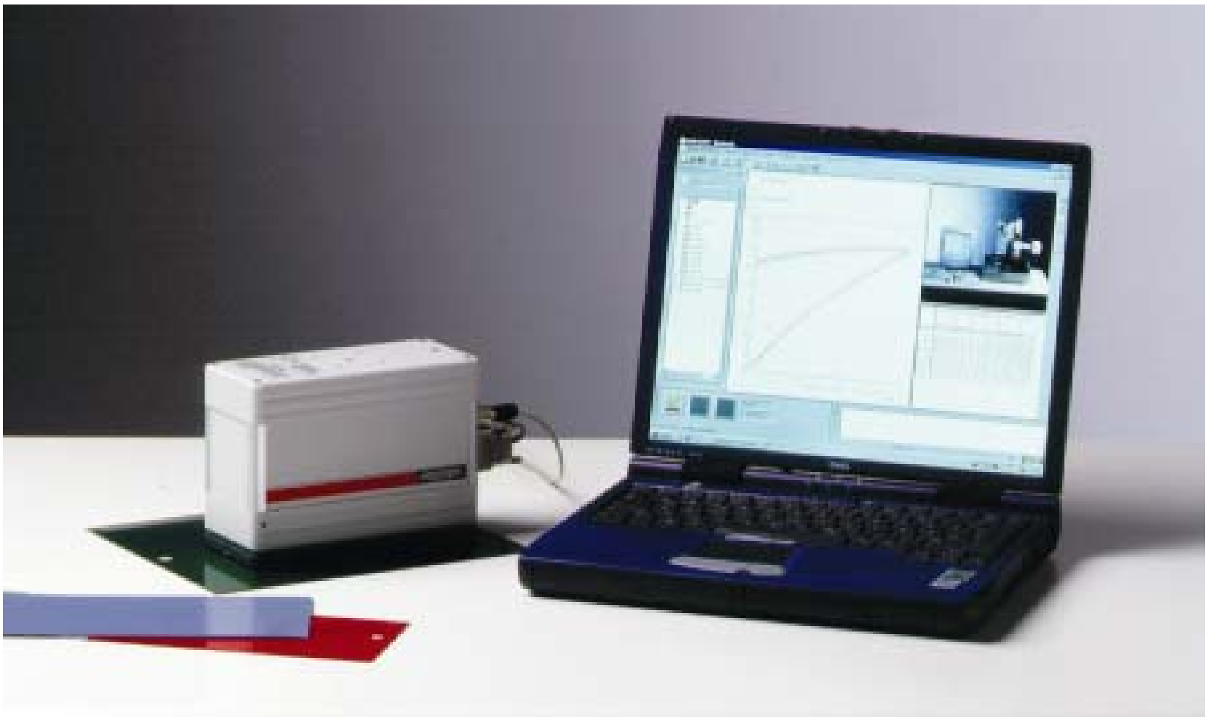

Image 1: Fischerscope H100 C- Measuring Head H100SMC with PC
(Photo: H. Fischer GmbH –Sindelfingen)

For testing metallic materials and material coatings DIN 50359 and ISO 14577 define the **micro range** test load with an upper limit of $F < 2N$ and the indentation depth with a lower limit of $h > 0.000\ 2$ mm / 4,5 /. ISO 14 577 expands the application range versus DIN 50 359 for the instrumented indentation test for test loads up to 30,000 N and for indentation depths $h \leq 0.000\ 2$ mm (nano range).
Table 1 summarizes the specified limits for the load and distance measurements in the standards mentioned.

Table 1: Limit deviations of the test load; Resolution of the limit deviation of the distance measurement system according to /1, 2/

Application range	Limit deviation of the test load	Limit deviation of the distance measurement system	Resolution of the distance measurement system
$F \geq 2$	± 1 %		
$0.1 \leq F < 2$	± 1.5%		
$0.001 \leq F < 0.1$	± 2.5%		
Macro range		± 1 % of h	≤ 0.00 1 mm
Micro range		± 1 % of h	≤ 0.000 01 mm
Nano range		± 0.000 002 mm	≤ 0.000 001 mm

Comparing the instrument parameters of the measuring head H100SMC with the requirements of the standards indicates that the Fischerscope H100C is qualified for a large range of applications. The available extensive test measurements confirm this position. The limit deviations determined for several nominal test loads are compiled in Table 2. The permissible test loads are clearly under-run down to test loads of 5 mN. Even for the nominal test load of 1 mN, the deviation from the actual value with 1.8% is below the requirements of the test standard.

Table 2: Measuring head H100SMC – measured limit deviations for various test loads

Test load - nominal value	Test load - actual value	Limit deviation
1000 mN	999.96 mN	0.004 %
100 mN	100.004 mN	0.004 %
10 mN	10.014 mN	0.14 %
5 mN	5.018 mN	0.36 %
1 mN	1.018 mN	1.8 %

The progression of the measured load/depth plot for a nominal load of 10 mN shown in Figure 2 is typical for the nominal test loads examined in Table 2.

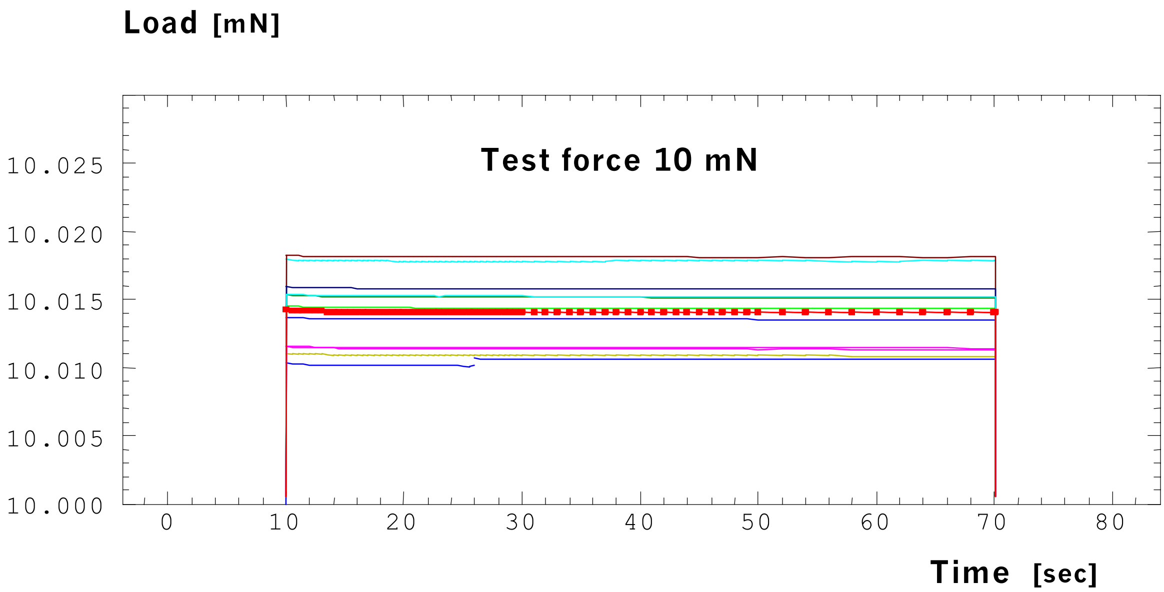

Figure 2: Load/time progression for 10 measurements at the nominal test load of 10 mN

F_m = 10.014 mN - F_{min} = 10.010 mN - F_{max} = 10.018 mN

The load/indentation depth plots measured for the test load of 10 mN on glass, type BK7 and shown in Figure 3 (Number of measurements: 5) indicate that the measurements with the introduced measurement technology could be performed precisely and reproducibly down to the nanometer range.

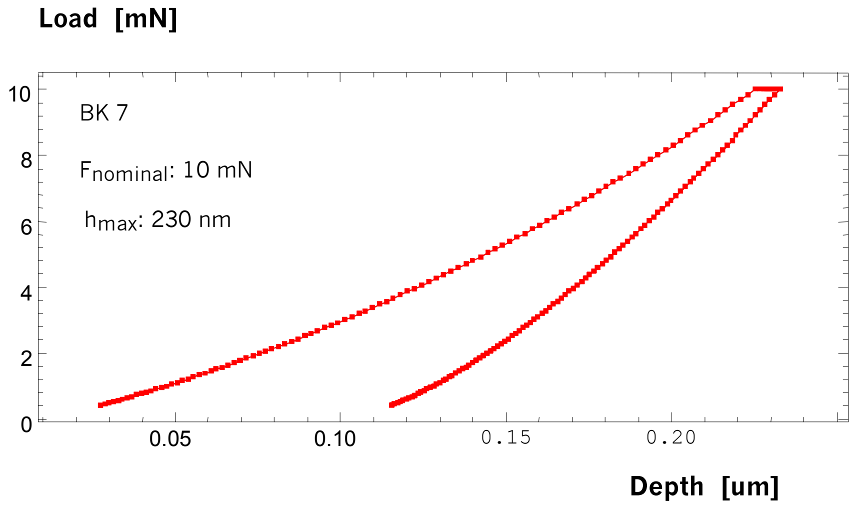

Figure 3: Load/indentation depth plot on Glass BK7 for the nominal test load of F =10 mN

In addition to the accurate and synchronous measurement of test load and indentation depth, the measurement uncertainty, and with it the useful range of application for the instrumented indentation test, depends to a large degree on the accuracy of the determination of the zero point for the load/indentation depth progression.
According to DIN 50 539, the measurement uncertainty of the zero point may not exceed 5 nm or 1% of the indentation depth (the larger value applies). The permissible level for the micro/nano hardness border zone is, therefore, 2 nm.
ISO 14 577 specifies the measurement uncertainty for the zero point with max 1% for the macro and micro ranges. Figure 4 reflects the results for the check measurements for the zero point determination on a hard Si sample and a soft PMMA sample using the Fischerscope H100 C. For the applied test load of 100 mN, the max. indentation depth for the Si sample is 630 nm, requiring a test uncertainty for the zero point determination of 6 nm. For the PMMA sample, the max. indentation depth for the test load of 10 mN is about 1.22 µm and the permissible measurement uncertainty for the zero point, is therefore, 12 nm. As Figure 4 indicates, the method used for the zero point determination stays significantly below the permissible measurement uncertainties.

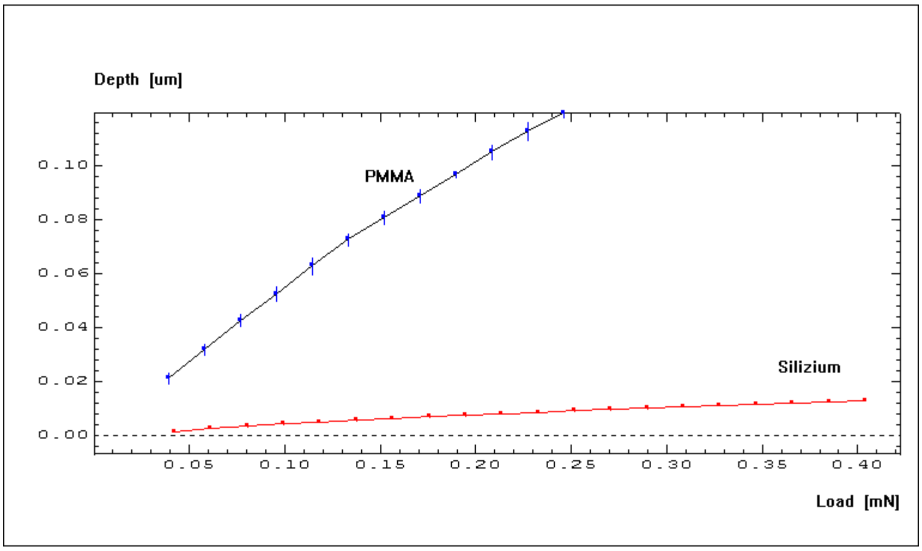

Figure 4: Fischerscope H100 C- Zero point determination on a hard and a soft material

3. Testing and evaluating coatings based on their use

The application behavior of the coating/substrate composite formed through coating is a system property that is based on the interaction of the property profiles of the coating and the substrate materials. To a large degree, the use behavior of coated products is determined by the mechanical properties of the coating. Coatings used in technical applications are very hard or also relatively soft and the coating thicknesses may be as thin as a few μm or even less than 1 μm. Increasingly, applications call for extremely thin coatings in the nano range.
Following is a report about the use of the mobile measuring head H100SMC for the mechanical characterization of coated large-area and compact specimens.

Paint and lacquer coatings

The hardness/depth plots measured for various test loads and shown in Figure 5 are characteristic for lacquer coatings on a very hard substrate material and for very low load application speeds (≤ 1 mN/ s). The hardness is greatest at the surface and then drops off to various degrees corresponding to the test load. If the test load is too great, the influence of the substrate material hardness will be noticeable. Beginning with an indentation depth of about 3.5 μm (1/10 of the coating thickness), the hardness reading increases again. A mixed hardness is determined for the used test load of 100 mN. Thus, selective hardness values for lacquer coatings are comparable only by supplying information about the test load or the indentation depth. The influence of the load application speed always needs to be taken into account as well.

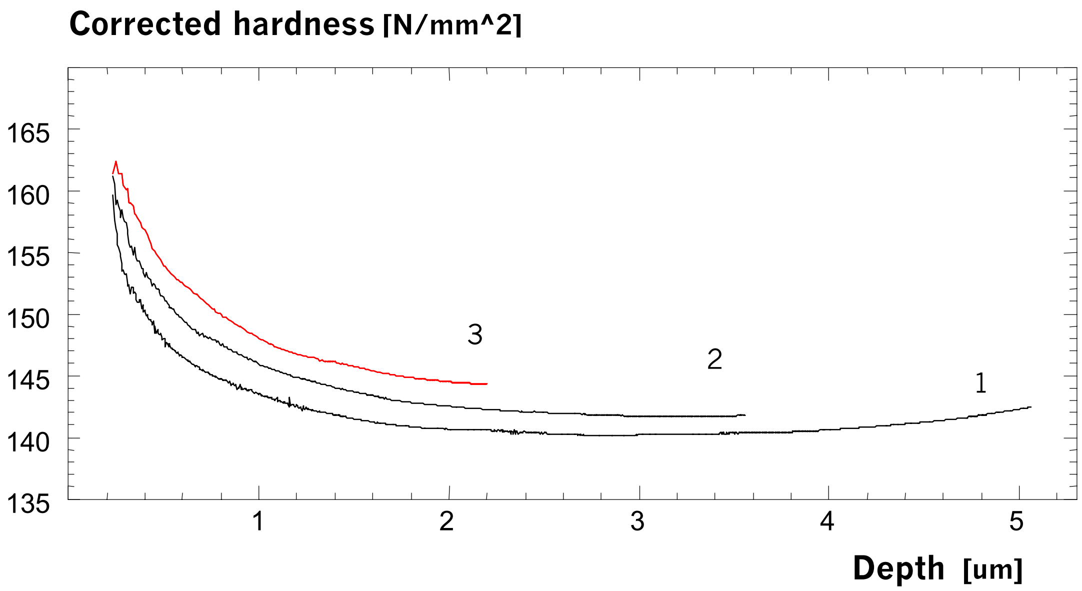

Figure 5: Martens hardness/indentation depth plot of a clear lacquer coating for various test loads; load application speed = 1 mN/s

The load application speed has a significant influence on the hardness as well as on the other characteristic quantities of the instrumented indentation test as is indicated by Figure 6 and the characteristic data in Table 3. When increasing the load application speed, the hardness at the surface increases, and then drops continuously. This fact did not receive sufficient attention in the past. When revising DIN 55676 *Lacquers and Coating Materials - Universal Hardness of Coatings,* provisions should be made that ensure the determination of comparable characteristic data.

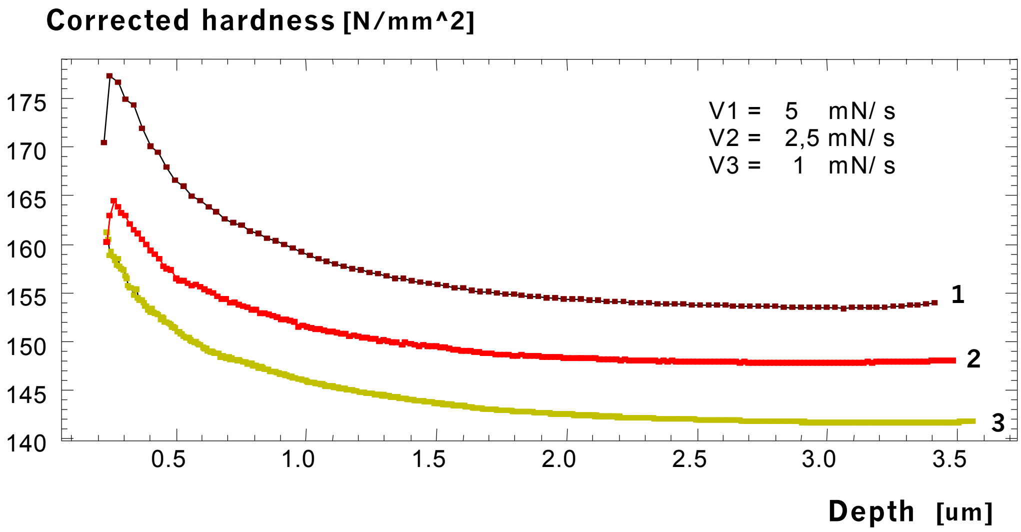

Figure 6: Martens hardness as a function of the indentation depth, determined for various load application speeds

Table 3: Mechanical properties of a lacquer coating for various load application speeds – test instrument Fischerscope H100 C, test load 50 mN

v /mN / s /	HM corr. / N/mm²/	W_e/ W_{tot} / % /	Cr 1 / % /	Cr 2 / % /	h_{max} / µm /	Y_{HM} / GPa /	HM(F) 20 mN /N/mm²/
25	92.4	32.9	12.0	-26.9	4.36	2.42	104.5
10	82.8	32.2	9.4	-25.3	4.61	2.48	92.1
2.5	73.7	31.2	3.8	-12.9	4.89	2.91	79.5
1.0	65.3	25.9	2.5	-12.1	5.22	2.98	71.2
0.5	60.9	25.3	1.4	- 7.8	5.40	3.07	65.7

Chromium-coated products

Chromium coatings find a variety of uses in technical applications. Until now, the capabilities for evaluating the mechanical properties of chromium-coated semi-finished and finished products have been very limited.
Measurements on chrome-plated coatings performed with the mobile measuring head H100 SMC prove that a new measurement device is available for quality control even for this area of application. Using a simple adapter, the measuring head can be positioned without much effort at the desired test location for measurements on site, even with curved surfaces (Figure 7).

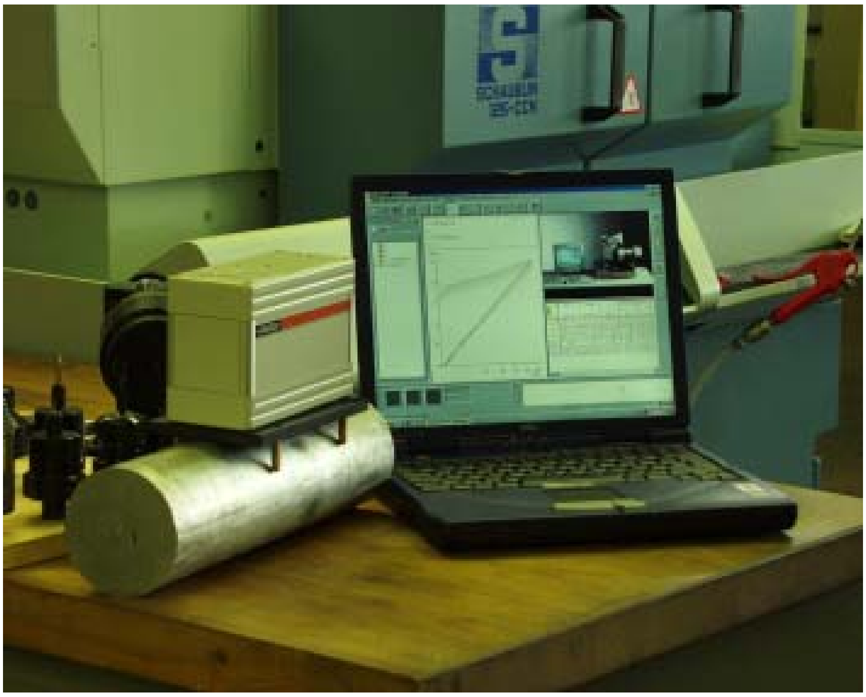

Figure 7: Martens Hardness Measurement of a chromium coating on site using the Fischerscope H100 C

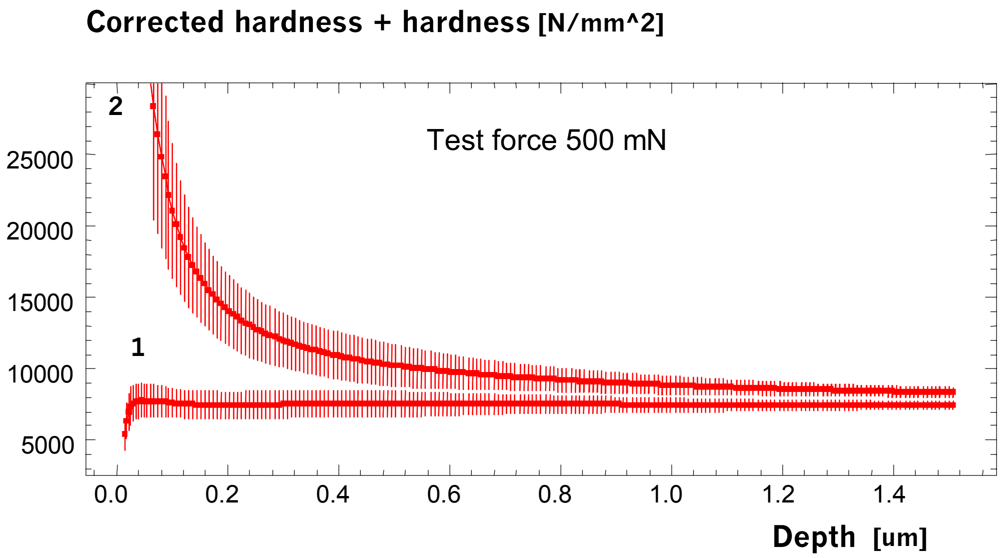

Figure 8: Martens Hardness on a chromium-coated component using the Fischerscope H100 C – with (1) and without (2) indenter correction

When evaluating the test results from 5 measurements, it should be noted that as a rule chromium coatings are very inhomogeneous. In this case, the occurring deviations are not the result of the application conditions on site. A check measurement of the zero point resulted in a deviation of max. 2 nm. Thus, for the current indentation depth of 1.46 µm, the limit deviation is at max. 0.15 %.

The hardness progressions shown in Figure 8 also document well the importance of regular check measurements and the need for taking the geometry of the indenter that is being used into account for measurements with small indentation depth ranges.

Table 4: Test results for a chromium coating (Figure 7) – Test load 500 mN

	Martens hardness *HM* N / mm²	Welast / Wtot %	Indentation creep %	Modulus of indentation N / mm²
Mean value	7398	31	1.1	268
Number of measurements	10	10	10	10
Standard deviation	271	0.53	0.12	16
Coefficient of variation	3.7	1.7	10.8	5.9
Range	701	1.5	0.4	49
Range/%	9.5	4.8	34	18.5

4. Conclusion

From the available measurement results of which only extracts have been presented here, it can be said in conclusion that the measurement technology implemented in the mobile measuring head H 100 SMC for the Fischerscope H100C with its various configurations opens entirely new applications.

In addition to high-resolution and reproducible measurements with small test loads and indentation depths, the prerequisites have been created for tests on large-area and on compact specimens even on site. Using the very variable and quick positioning ability of the measuring head on the desired test location, large areas can be tested and evaluated efficiently in sections or at stress-critical areas. Expanded fields of application in quality control can be expected for the mobile measuring head.

5. Bibliography

/ 1 / Product information Fischerscope H100C
Helmut Fischer GmbH +Co. KG - Sindelfingen

/ 2 / G. Michalzik Mechanische Schichtcharakterisierung
HTM - Härterei-Technische Mitteilungen 56 (2001) 6 S. 410-416

[Mechanical Coating Characterization

HTM Hardness Technical Messages 56 (2001) 6 P. 410 – 416]

/ 3 / Mechanical characterization of hard material coatings –
Using the example of a 2.5 μm TiN coating
Application report of Helmut Fischer GmbH +Co. KG

/ 4 / DIN 50359-1 Prüfung metallischer Werkstoffe - Universalhärteprüfung
Teil 1: Prüfverfahren Beuth-Berlin
[Measurement of metallic materials – Universal Hardness Test
Part 1: Test method Beuth-Berlin]

/ 5 / ISO 14 577-1 Metallische Werkstoffe - Instrumentierte Eindringprüfung zur Bestimmung der Härte und anderer Werkstoffparameter
Teil 1: Prüfverfahren Beuth-Berlin
[Metallic materials – instrumented indentation test to determine the hardness and other material parameters
Part 1: Test methods Beuth-Berlin]