

EVALUATION OF MECHANICAL PROPERTIES IN THE NANOMETER RANGE – PRECISION AND TRUENESS

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The determination of mechanical properties of ultra-thin films or very small components becomes more and more important due to the increasing number of applications of such devices. However, an accurate mechanical testing of coatings with a thickness down to some nanometer is still a challenge, despite the improvements in mechanical property testing. The instrumented indentation test (IIT, Nanoindentation) is an advanced technique for mechanical property determination. The principle of IIT is described in the recently issued standard ISO 14577 [1]. While the IIT is very well described, studies about the certification of reference materials, precision and trueness of this method in the nanometer thickness range are relatively seldom [2,3].

For evaluation of IIT and certification of coating/substrate reference systems the realisation of an accuracy experiment (inter-laboratory comparison) according to ISO 5725 1-5 [4] was proposed in VAMAS TWA 22 (Mechanical Property Testing of Thin Films and Coating). For comparison and for evaluation of trueness of the data achieved by IIT, surface acoustic wave method (SAW, dispersion of ultrasonic surface waves) according to DIN 50992-1 [5] was used. In VAMAS TWA 22 project 3 BAM in Germany, AIST in Japan and NIST in USA have agreed to perform a first inter-laboratory comparison (IIT and SAW) on prototype reference coating/substrate systems prior to the final inter-laboratory comparison. The aim of this study is to present the results this first comparison.

The prototype reference samples (glass BK7; Cr film (20 nm) on glass BK7; SiO₂ film (1 µm) on glass BK7; Cr film (20 nm) on SiO₂ (1 µm) on glass BK7; TiO₂ film (1 µm) on glass BK7; Cr film (20 nm) on TiO₂ film (1 µm) on glass BK7) were prepared by SCHOTT Glas Germany. All experiments have been conducted according to ISO 14577 using the commercially available testing instrument Nano Indenter XP (MTS). The test force, the displacement measuring device and the machine compliance have been calibrated according to this standard. According to ISO 14577 the measured force/indentation depth data-sets must be corrected for the thermal drift, the machine compliance and the zero point. After correction of the thermal drift, the data-sets were corrected with respect to compliance under the applied maximal test force. This was done by using the standard MTS software and the software INDENT ANALYSER [6]. This software allows the evaluation of the measured raw data independent from the standard software and provides some additional corrections.

After definition of the standard measurement method, IIT measurements have been conducted by all participating laboratories. Using this experimental data set, the repeatability and the reproducibility for all laboratories have been calculated. For the investigation of trueness of IIT method the results of SAW measurements have been accepted as reference values. The measure of trueness was expressed as difference between the IIT results and the accepted reference values.

[1] ISO 14577 1-3 Metallic materials – Instrumented indentation test for hardness and materials parameter – Part 1–3

[2] Determination of Hardness and Modulus of thin Films and Coating by Nanoindentation – Indicoat Final Report. NPL Report MATC(A), Teddington (2001)

[3] K. Herrmann, N.M. Jennett, S. Kuypers, I. McEntegart, C. Ingelbrecht, U. Hangen, T. Chudoba, F. Polenz, F. Menelao: Investigation of the properties of candidate reference materials suited for calibration of nanoindentation instruments. Z. Metallkunde 94-1 (2003) 802-806.

[4] DIN 50992-1 Thickness measurement of coatings and characterising of surfaces with surface waves – Part 1: Evaluation of elastic constants, mass density and thickness of layers by laser- induced ultrasonic surface waves.

[5] ISO 5725 1-5 Accuracy (trueness and precision) of measurement methods and results

[6] ELASTICA was developed at TU Chemnitz and is now a product of ASMEC
(trial version at www.asmec.de)