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Pico litre volume measurement with a laser focus sensor on the nano measuring machine NMM-1

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Abstract
A novel approach of measuring small volumes is made in our project. The goal is to utilise the Nano Measuring Machine NMM-1 in combination with a laser focus sensor to determine the volume of small water droplets. The droplet surface is scanned by the focus sensor with picometre resolution. Based on the shape of the scan lines, the actual volume of the droplet can be calculated.
However, due to the small distance between the water surface and the glass carrier, the laser focus sensor signal was distorted by undesired reflections from the glass carrier the droplets are deposited on.
To reduce these undesired reflections, the BK7 carrier was anti-reflection coated. When a SiO_2 layer of appropriate thickness is deposited on the carrier, the reflectance at the water – glass interface can be considerably lowered. This drastically improves the sensor signal, thus enables the correct measurement of the droplets' shapes and hence the calculation of their volume.

Keywords: precision volume measurement, laser focus sensor, nano measuring machine, anti-reflection coating

1. Introduction

For research fields such as microfluidics, medicine, nanotechnologies etc. the exact and traceable determination of volumes in the range of microliters and smaller is a challenging and important task. There are approaches using secondary electron microscopy or atomic force microscope for dried drops of solutions [1], gravimetrical [2] as well as photometric [3] methods.

In this paper a new approach is described which utilises the Nano Measuring Machine NMM-1 [4] and a laser focus sensor LFS [5] for a geometrical determination of the volume of small water droplets.

First, the measurement principle and the problems arising from the small distance between the water surface and the water – glass boundary and the following focus sensor signal distortions are described. After that the attempts are explained that were made to reduce these undesired reflections. Finally the simulation and fabrication of the anti-reflection coating of the glass carrier is described, which lead to a significant improvement of the sensor signal.

2. Measurement principle

For a geometrical determination of their volume, droplets of water are applied on a glass carrier made of BK7 and their surface is scanned by a LFS (figure 1). Based on the shape of the scan lines, the actual volume of the droplets can be calculated.

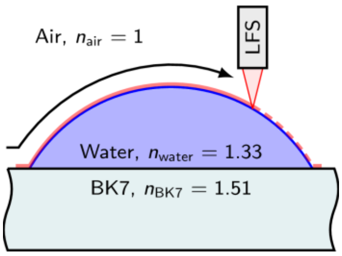

Figure 1. Scanning the shape of a water droplet on a glass carrier by a laser focus sensor (LFS).

The LFS used as the probe in these measurements is realised on the basis of a laser hologram unit with a fixed objective lens. It includes a CCD camera to allow the user to see the laser spot of the focus sensor and the surrounding surface during the measuring process.

The LFS is mounted as the probe in the NMM-1 which is a coordinate measuring machine with a 20 pm resolution and a 25·25·5 mm^3 measuring volume [4].

The position of the sample carrying corner mirror is measured by three interferometers and two angular sensors in six DoF, adhering the Abbe principle in all three directions of space (figure 2).

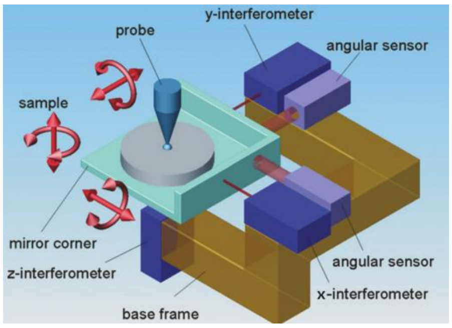

Figure 2. Basic principle of the NMM-1 [5].

With this measuring setup, the glass carrier is traversed underneath the LFS. The position of the carrier is controlled in such way that the focus spot of the LFS is on top of the water surface at any time. As the position of the carrier can be determined with picometer resolution, it is possible to measure very small drops which are unsuitable for e.g. gravimetrical attempts.

2.1. Measuring problems for very small droplets

Due to the small distance between the water surface and the BK7 carrier, focus errors occurred when measuring small droplet heights below 30 µm. The cause of the focusing problem is the comparatively high reflectance of the water–glass interface

($R = 0.4$ %) compared to the reflection on the water–air interface ($R = 2$ %). This lead to undesired reflections from the water–glass boundary which distorted the primary focus signal from the droplet's surface. The consequence of these distortions was the faulty measuring of the drop height, making the measurement of drops lower than 30 µm impossible.

Thus, it was necessary to reduce the reflectance between the water and the glass carrier.

2.2. Matching the refractive index

The first attempt to reduce the undesired reflections from the water–glass boundary was to use a carrier made of a material with a refractive index similar to water ($n_{\mathrm{water}} = 1.33$), since the reflectance depends on the difference of the refractive indices. Possible materials are e.g. sodium fluoride (NaF, $n_{\mathrm{NaF}} = 1.32$) or magnesium fluoride (MgF_2, $n_{\mathrm{MgF2}} = 1.38$). However, the main drawback of these materials is that they are either water-soluble or porous, which makes them unsuitable for this specific task.

3. Anti-reflection coating

The remaining solution for reducing the undesired reflections is an anti-reflection (AR) coating of the BK7 carrier. For a single Wavelength this can be achieved by a single AR layer of appropriate thickness and refractive index [6].

As the thickness of the water film can be considered large compared to the wavelength of the LFS ($\lambda = 650$ nm), the AR task can be reduced to the AR coating of a substrate (BK7) towards a medium (water) of infinite thickness. This task can be solved by a single layer of a thickness

$$d_{\mathrm{AR}} = {}^{\lambda}/_{4} \tag{1}$$

and a refractive index of

$$n_{\mathrm{AR}} = \sqrt{n_{\mathrm{BK7}} * n_{\mathrm{water}}} \tag{2}.$$

For the given conditions this results in $n_{\mathrm{AR}} = 1.42$ and $d_{\mathrm{AR}} = 114$ nm. If these values are met exactly, a reflectivity of zero can be achieved at the desired wavelength.

3.1. Simulation results

The properties of the AR layer in the previous section are ideal values. For an optimal AR coating, a material must be used which's refractive index matches the theoretical value best. A proper material is SiO_2 with $n_{\mathrm{SiO2}} = 1.46$. As the refractive index does not match the theoretical value exactly, the adjusted layer thickness was computed to 111 nm using the matrix method [6] and a minimum search algorithm. With this AR layer, the reflectance can be lowered to 0.078 %. Figure 3 shows the simulated reflectance of the AR coated BK7 carrier.

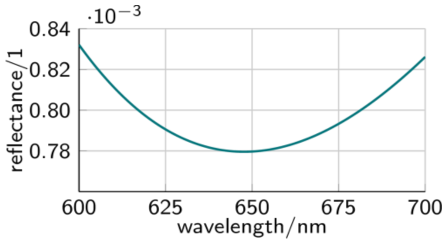

Figure 3. Simulated reflectance of the AR coated BK7 carrier.

4. Experimental results

With the AR coated carrier it is possible to measure also very small drop heights without signal distortions from the water–glass boundary. Figure 4 shows an exemplary cross section of a droplet without any observable focus errors.

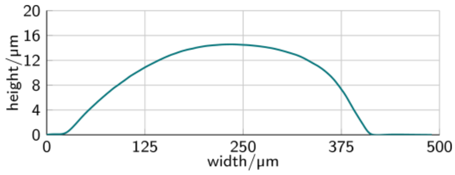

Figure 4. Cross section of a droplet with a height <30 µm on the AR coated BK7 carrier. There are no distortions in the focus signal observable.

The measured shape of some drops and their calculated volumes are shown in figure 5.

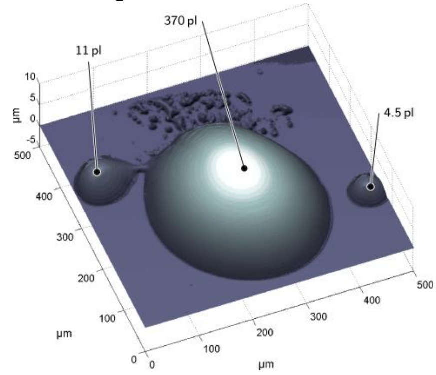

Figure 5. Examples of measured droplets and their calculated volumes.

5. Conclusion and Outlook

When measuring the shape of small water droplets on a BK7 glass carrier using an LFS on the NMM-1, focus errors occurred due to reflections from the water–glass interface, making a correct volume measurement impossible. Reducing the reflectance of this interface by matched refractive indices is not possible, as proper carrier materials are either water-soluble or porous and therefore cannot be used. With a single layer AR coating, the reflectance of the BK7 carrier could be considerably lowered from 0.4 % (uncoated) to 0.078 % (coated). This way it was possible to measure droplets with a height substantially smaller than 30 µm and calculate their volume based on the scan lines, enabling traceable volumetric measurements in the picolitre range for pipette calibration and similar purposes.

Future measurements will include optimised climate conditions (e.g. high humidity) to reduce the evaporation and the resulting shape modification of the water droplets during the measuring process.

References

[1] Sparks C M, Fittschen U E A and Havrilla G J 2010 Picoliter solution deposition for total reflection X-ray fluorescence analysis of semiconductor samples *Spectrochimica Acta Part B: Atomic Spectroscopy* **65** 805-811

[2] ISO 8655–6:2002 Piston-operated volumetric apparatus - Part 6: Gravimetric methods for the determination of measurement error. *ISO, Geneva*

[3] McMillan N, O'Neill M, Smith S, Hammond J, Riedel S, Arthure K and Smith S 2009 Reliable TLDA-microvolume UV spectroscopy with applications in chemistry and biosciences for microlitre analysis and rapid pipette calibration *Journal of Optics A: Pure and Applied Optics* **11** 054016

[4] Jäger G, Manske E, Hausotte T and Büchner H-J 2009 The Metrological Basis and Operation of Nanopositioning and Nanomeasuring Machine NMM-1 *tm-Technisches Messen Plattform für Methoden, Systeme und Anwendungen der Messtechnik* **76** 227-234

[5] Manske E, Hausotte T, Mastylo R, Machleidt T, Franke KH and Jäger G 2007 New applications of the nanopositioning and nanomeasuring machine by using advanced tactile and non-tactile probes *Measurement Science and Technology* **18** 520

[6] Yeh P 1988 Optical waves in layered media *John Wiley & Sons, New York*