Microplasticity (MP) is one of the parameters characterizing viscoelastic behavior of synthetic polymers [1,2]. MP is defined as the normalized ratio between the width and depth of the indent from microhardness tester after elastic recovery (Figure 1). In the past decades, MP was scarcely measured due to experimental difficulties [3]. Nevertheless, modern microscopic techniques made MP measurement feasible. Our preliminary results have showed that microscopically measured MP values are in a good agreement with material properties [3]. In this contribution we focus on four microscopic methods which can measure MP, i.e. which yield 3D surface maps with submicrometer accuracy, especially in Z-axis direction.

We tested the precision of the following four microscopic techniques: (i) wide-field light microscope with motorized Z-stage and software for combining Z-stack images into 3D-surface maps (DM6000 M, Leica), (ii) digital microscope, which provides 3D-surfaces with higher precision (VHX-1000, Keyence), (iii) standard SEM microscope (Vega Plus TS 5135, Tescan), in which the depth information is calculated from precisely tilted micrograph as described by Lawn et al [2], and (iv) advanced SEM microscope (Quanta 200 FEG, FEI) with eucentric stage and Stereo software (Scandum software with Stereo module, Olympus), which enables the user to create stereoscopic images and to compute the height of image points from two images tilted to small angle (typically +/- 3 degrees).

The testing samples were two polymers (two types of ultrahigh molecular weight polyethylene – UHMWPE – with different thermal treatment and plasticity) and one metal (B2-ordered intermetallic alloy Fe - 41 at.% Al). MP of each sample was determined from at least five indents, which were left to equilibrate for 2 days and subsequently investigated by all above described methods.

Typical outputs from 3D microscopic methods are shown in Figure 2. With exception of standard LM microscope, all methods were in agreement with theoretical prediction that metal sample is >75 % plastic and polymer materials exhibit MP values within 20–30 % [3]. Final comparison of all results (Figure 3) suggested the following precision of the methods: standard LM microscope << digital microscope = SEM microscope (calculation according to [2]) = SEM microscope (calculation from stereo images using commercial software).


Fig. 1: A schematic illustration of the principles of MP determination by means of Vickers pyramid indenter and the MP calculation formula [1].

![Diagram of indenter and sample](image)

\[
\alpha = 136.00^\circ \quad \text{(angle between opposite faces)}
\]

\[
\beta = 148.11^\circ \quad \text{(angle between opposite edges)}
\]

For completely elastic materials: \( h/a = 0 \)

For completely plastic materials: \( h/a = 2/7 \) (\( h/a = \cot^{-1}(148.11^\circ/2) \))

\[
MP = h/a \times 7/2 \times 100\%
\]

Fig. 2: A - SEM micrograph of 50° tilted indent, B – micrograph of the indent obtained by means of digital microscope, C – typical depth profile of an indent used for MP determination.

![Images of micrographs](image)

Fig. 3: The overall results obtained by all 3D-microscopic methods. Note: remelting = thermal treatment above melting temperature, annealing = thermal treatment below melting temperature.

![Graph of microplasticity](image)