The Examination of Photopolymer-based 3D Printed Products in the Case of Pinpoint Loading

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***Abstract*— We present a new mounting unit for Dynamic Mechanical Analysis (DMA) equipment, suitable for measuring microhardness and indentation, and its usability on photopolymers. We used a Vickers-type microhardness indenter, and examined the dimensions of the indented area and the loading force.**

***Keywords*—photopolymer, pinpoint load, measurement of indentation, Vickers, DMA**

1. INTRODUCTION

Nowadays polymers are more and more widely used in all facets of life. They can replace metals in numerous areas of use, due to their adequate strength, low density, good corrosion resistance and vibration damping ability, but their mechanical behaviour can be considerably different from that of metals. In the case of polymers it is not enough to examine quasi-static mechanical characteristics (tensile and flexural tests) because they react to long-term loading with continuously increasing deformation; this is called creeping. Therefore, it is an important rule for designers that they should design parts not for lifetime but maximal possible deformation. Creep is most widely determined with tensile and flexural tests, but torsion and compression loading is also used. The latter can be loading on area or a pinpoint load [1, 2].

As additive technologies develop, materials and procedures offer more possibilities to designers and users alike. A great advantage of products manufactured this way is that their weight can be easily adjusted with the degree of filling and the product can have almost any geometry. Polymer-based 3D printed products are not only prototypes nowadays but are also used as functional products (e.g. dental implants), therefore it is important that as many technical parameters of these materials are revealed as possible so that their areas of use can be accurately determined [3, 4].

The hardness of materials is an important characteristic that helps designers select the right material. It is defined as the resistance of a solid material to penetration by a body. Pinpoint compression loading can be examined very well with a hardness test. Depth-sensitive (dynamic) hardness testing (depth sensing indentation – DSI) was developed while the advantages of hardness testing (simplicity, little use of material) retained. This test can characterize mechanical properties dynamically. During this process the measuring body is pushed into the surface at constant speed or with constant force, and then removed. The impression depends on the properties of the material and the geometry of the indenter, and various characteristics can be determined from it, such as dynamic hardness, conventional hardness, modulus of elasticity, and elastoplastic mechanical properties. The geometry of the impression depends on the depth of indentation, which in turn depends on the loading force, and the time of loading, and of course, environmental parameters, such as humidity and temperature [3].

The advantage of depth sensing indentation is that measurements can be made in the millimetre, micrometre and nanometre range as well. This facilitates the testing of various thin films, or new materials produced by nanotechnology (e.g. nanocomposites), even with very little loading forces (in the case of nanohardness testing, it can be 0.01 mN). This procedure may be suitable for the testing of outer, even coated layer of 3D printed polymer-based products. A disadvantage is that the measurement results are always local, and can be very different Also, there are two important phenomena to be mentioned. Next to the indenter, at the side of the indented area, the surface can sink (sinking-in), or can be raised because material is pushed out (pilling-up), and these can cause inaccuracies (Fig. 1). For this reason, several tests have to be performed for accurate results [2].

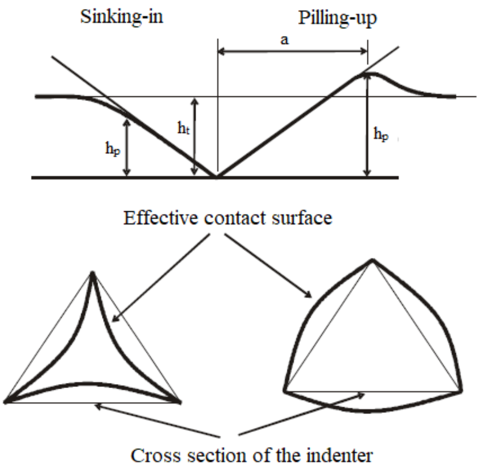

Figure 1. Sinking-in and pilling-up [2]

According to the literature we analysed, [6-8] this procedure, combined with creep tests, is used successful on polymer materials. The geometries of the indenters are the same as used in the case of static hardness tests, but mostly the Vickers and Berkovich types are used. Usually, finite element analysis is performed as well, to support the measurement results.

The literature [6-8] shows that although this method is spreading, tests are performed almost exclusively at room temperature. Objects used in everyday life are exposed to different temperatures and humidity; therefore we decided that tests should be performed in a wide range of temperatures. In order to provide the small loading forces and constant temperature required for the test, we designed a mounting unit suitable for pinpoint loading for a dynamic mechanical analyser (DMA), with which short, long and also cyclical tests can be performed.

2. THE TESTS

2.1. The mounting unit designed

We designed the mounting unit for a TA Instruments (USA) Q800 device. The analyser can perform various tests (tensile, compression, flexural and shear tests) in a wide temperature range (-145 to +600 °C). It has a frequency range of 0.01 Hz to 200 Hz, and the maximum load is 18 N. The device can determine the mechanical and viscoelastic properties of various materials. Both constant and periodic loading can be applied [9].

We designed the mounting unit based on an existing fixture used for compression tests. We tested the two designs we liked best with finite element analysis to see what deformations occur. The two designs were the one where the indenter is fastened in the middle of the fixture, and the “horse race track” design (this allows the positioning of the indenter). Fig. 2 shows that in the case of the horse race track design, in an extreme position, at a load of 18 N, the frame deforms as well, which can destroy the air bearing of the DMA and the mounting unit itself, especially if deformation resulting from temperature change is also taken into account. Therefore, we selected as the final design the centrally positioned, gripped indenter design.

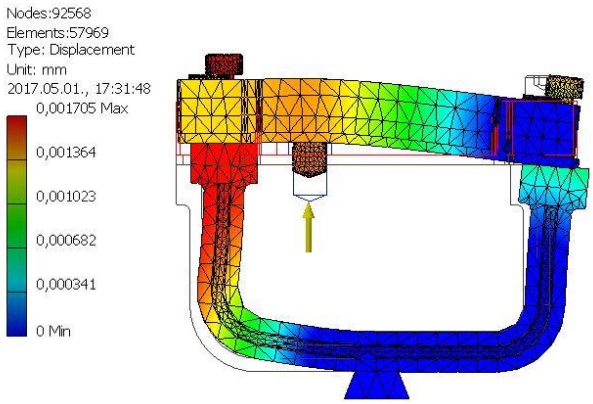

Figure 2. The finite element simulation results of the horse race track in an extreme position

We made the sample holder so that the sample can be positioned. This makes it possible to perform several measurements on a single sample. Thanks to the vice design, the specimen can be tightly gripped and so it does not move during the test. The final design can be seen in Fig. 3.

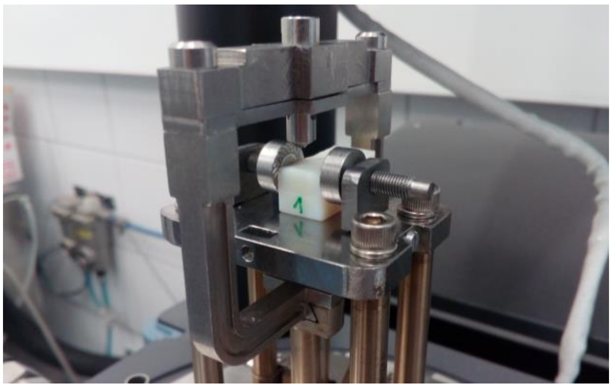

Figure 3. The fixture we designed and the gripped photopolymer specimen

2.2. The test program and the investigated materials

We tested two different kinds of photopolymers in the trial tests: RGD720 (FullCure 720) and RGD835 (Vero White Plus). The dimensions of the specimens were 10 mm x 20 mm x 9 mm. We performed the test on the 10 mm x 20 mm untreated (unpolished) surface, the top layer according to build direction. In the first stage, we increased loading force (F [N]) by 0.5 N in each step to 4 N, then in the second stage we used steps of 1 N and went up to 10 N, and finally we went all the way up to 18 N in steps of 2 N. We repeated all individual steps three times. This way we covered the force range of the DMA. In the tests, we used a Vickers microhardness indenter, which is a pyramid shape and is made of diamond. The geometry reduces dependence on loading force. The parameters measured on the negative mark of the indenter can be seen in Fig. 4, where *a* and *b* are the side length, d_1 and d_2 the diagonals and *h* the cone-height of the Vickers intender.

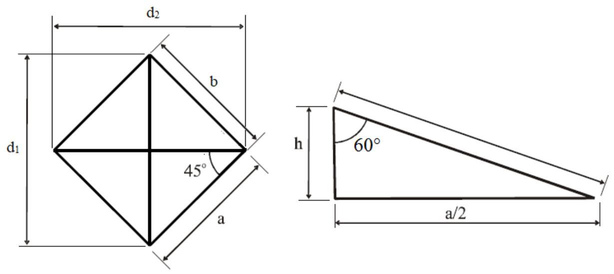

Figure 4. The impression of a Vickers indenter and its geometrical parameters [2]

The testing program consisted of the following steps: we heated the heat chamber to 35 °C, and then held this temperature for 3 minutes. Then we applied the loading force, which we held for 1 minute. After loading ceased, we analysed the indented area with an optical microscope (Olympus BX51M), and measured the diagonals of the indented areas (d_1 , d_2 [µm]).

2.3. Measurement results

As mentioned above, evaluation started with the microscopy examination of the indented areas. At a loading force of 0.5 N, the indented area could not be seen very clearly in the case of both materials. Above 1 N, however, the outlines of the rhombus-shaped indented area were clearly visible. We experienced pilling-up and sinking-in several times during the tests, these are shown by Fig 5.

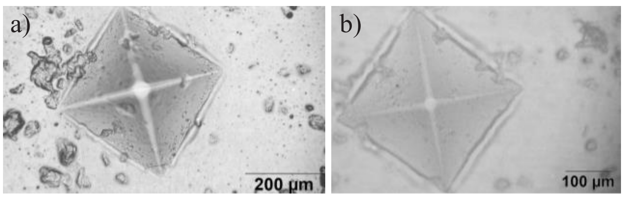

Figure 5. Pilling-up on the RGD720 (a) and sinking-in on the RGD835 material (b)

The microscopic images show (Fig. 6) that an increase in loading force enlarges the diagonals of the impression. The results show that the RGD810 material is softer; at the same load, the indenter left a larger impression on the surface of the specimen.

We examined whether the machine can push the indenter into the sample properly, perpendicularly to the surface. The diagonals of the impressions were averaged separately and displayed in one diagram (Fig 7.).

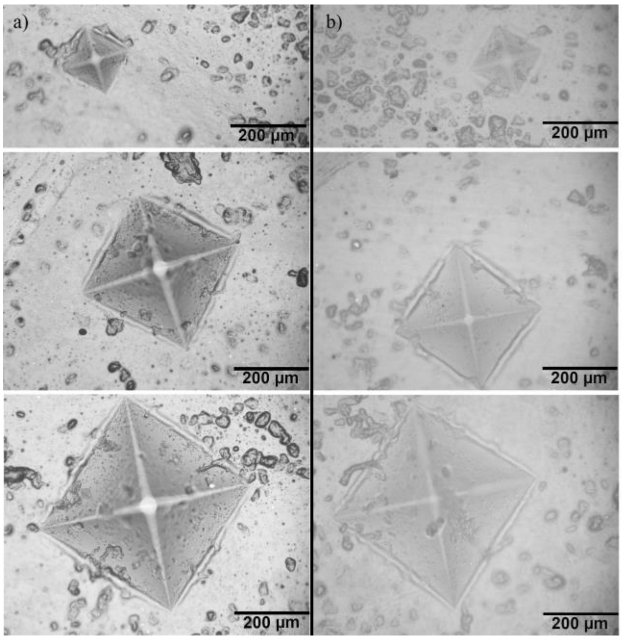

Figure 6. The impression on the RGD720 (a) and the RGD835 (b) materials at a loading force of 1 N (top), 6 N (middle) and 12 N (bottom)

It is visible that the points are on one line with little deviation. This proves that the impressions are symmetrical; therefore, the fixture can be used with indentation tests in a DMA. The largest standard deviation in the case of RGD720 is 18,595 µm, while in the case of RGD835, it is 13,218 µm.

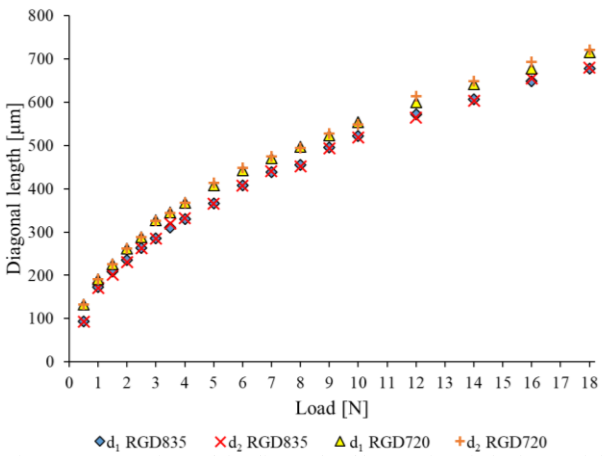

Figure 7. Comparison of the diagonals of impressions in both materials

As previously we proved that the impressions are symmetrical, the diagonals measured with a microscope can be averaged and the values can be displayed in a scatter plot as a function of loading force (Fig. 8). Also, if the diagonals are known, the projected area (A [µm^2]) of the pyramid created by the indenter can be calculated (1), and its change can be plotted as a function of the force (Figure 9).

$$A = \frac{d_1 \cdot d_2}{2}\ [\mu m^2] \tag{1}$$

In the case of both diagrams, we fitted a function of the same equation (2) on the points, which produced the highest determination coefficient (R^2). Its coefficients (*a, b, c*) can be determined with the method of least squares with the help of the measured series of points (Table 1).

$$f(x) = a \cdot (1 - e^{-b \cdot x}) + c \cdot x \tag{2}$$

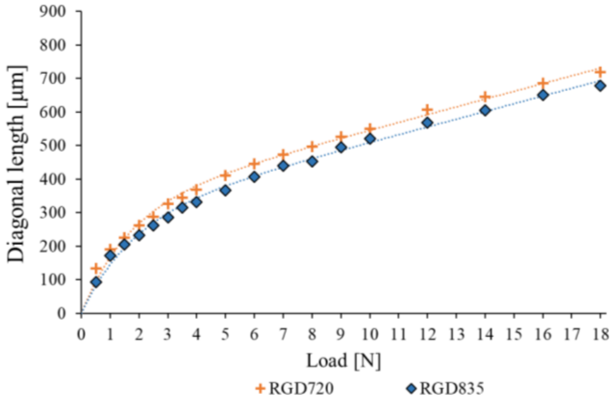

Figure 8. Average diagonals as a function of loading force

TABLE 1. THE COEFFICIENTS OF THE FITTED FUNCTION FOR THE DIAGONAL – FORCE (A) AND THE PROJECTED AREA – FORCE (B) CURVES

		a [μm]	**b [$\frac{1}{N}$]**	**c [$\frac{\mu m}{N}$]**	**R^2 [-]**
A	RGD720	314,74	0,61	23,131	0,994
	RGD835	280	0,57	22,983	0,995
		a [μm^2]	**b [$\frac{1}{N}$]**	**c [$\frac{\mu m^2}{N}$]**	**R^2 [-]**
B	RGD720	13848	0,45	13792	0,999
	RGD835	4504	0,42	12818	0,998

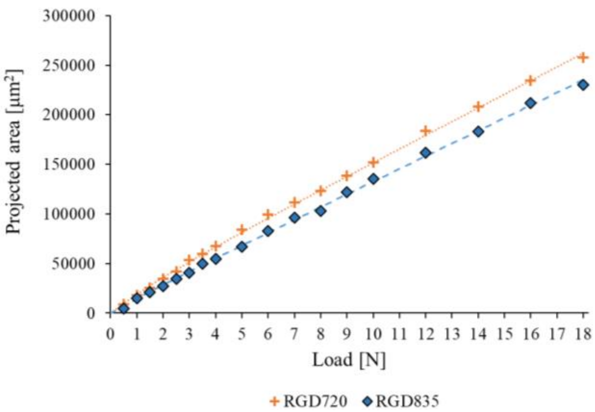

Figure 9. Projected area as a function of loading force

The function can be used well in the case of small forces too. It can be seen that the average diagonal–loading force curve has an initial power-like character, then after 7 N it increases nearly linearly. This is less visible on the projected area curve but when enlarged (Fig. 10), it shows that the linear character starts after 3 N.

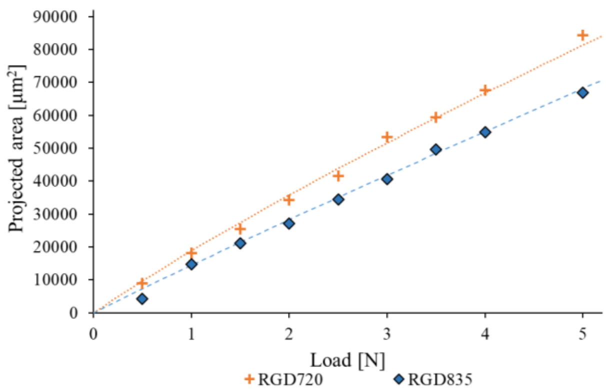

Figure 10. The projected area as a function of loading force, enlarged

CONCLUSIONS

The tests showed that the fixture we designed is suitable for depth sensing indentation tests with DMA equipment. This method can be used for the mechanical testing of the surface of prototypes or even finished products. This procedure can have an especially important role in materials development and in coating development, as important technological parameters can be determined in the case of thin layers, too. The method can also provide accurate information about the mechanical properties of the outer layer.

Thanks to the DMA equipment, the procedure can be used not only for static tests but also dynamic or even cyclic tests. In addition, tests can be performed in a wide range of temperatures.

In the case of longer tests, creep has to be expected, which is characteristic to polymers. In smaller sizes, creep can be more pronounced; therefore more detailed tests could be done in connection with this.

In the future the same series of tests as we did could be performed on specimens with a polished surface, and it could be examined whether any surface hardening occurs due to polishing, and also, what is the influence of the disappearance of surface defects.

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