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MECHANICAL CHARACTERIZATION OF ADVANCED CERAMIC MATERIALS USING NANOINDENTATION

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***Abstract*—**The paper aims to present the result obtained during mechanical characterization of ceramic materials. The characterization of the materials was realized using the G200 nanoindenter with the goal of fully characterizing the mechanical proprieties (hardness, and modulus).

***Keywords*—**ceramic materials, mechanical characteristics, nanoindenter

I. INTRODUCTION

INDENTATION is the technique in which a harder material commonly referred to as an indenter is used to deform a softer material[1].

The first test for hardness measurements was developed by the Swedish metallurgist Johan August Brinell. The Brinell method uses up to 30,000 (N) force applied using a spherical indented on the tested material [1].

Another method is the Rockwell Test, which uses a diamond cone to indent the tested material with forces the range of 600 (N) to 1500 (N) [1].

These methods although highly standardized and widely used are not suitable for new advanced materials and thin film materials.

Nanoindentation is now the standard tool for the mechanical properties measurement of all small scales, and it has even greater importance as a technique for of fundamental materials physics studies [2]. In nanoindentation small loads and tip sizes are used, so the indentation area may only be a few square micrometers or even nanometers [3].

In nanoindentation measurements, contrary to other hardness tests, the depth of the penetration of the tested material surface is determined as the load force is applied to the indenter [4]. Knowing the depth of the indentation and the geometry of the indenter tool allows the calculation of the area of indentation.

There are five main types of indenter tips, each with a different geometry for a variety of applications [5]:

1) *Berkovich*
2) Vickers;
3) Cube-Corner;
4) Cone;
5) Sphere;

For measurements of the mechanical properties on the nanoscale level, the most frequently used indenter tip for instrumented indentation testing is the Berkovich indenter tip [4].

In Fig. 1 the geometry of the Berkovich indenter tip is presented.

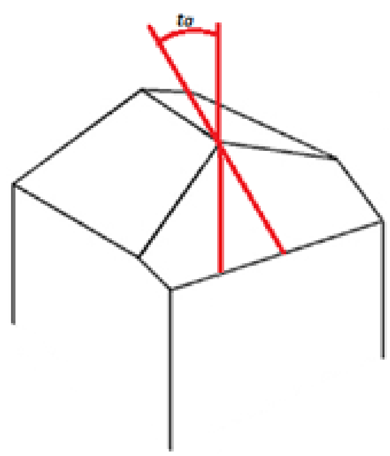

Fig. 1. Geometry of a Berkovich indenter tip

The shape of the Berkovich indenter tip consists of a three-sided pyramid that may be ground to a point in order tomaintain a self-similar geometry to very small scales [4]. The Berkovich indenter tip is suitable for most testing applications.

Some examples of Berkovich indenter tip applications include [5]:

1) Bulk Materials
2) Thin Films
3) Polymers (E' > 1GPa)
4) Scratch Testing
5) Wear Testing
6) Micro-electromechanical Systems (MEMS)

7) In-situ Imaging

For determining the hardness of materials using indentation techniques, the following formula is used [1]:

$$H = \frac{F}{A} \quad (1)$$

Where H is the calculated hardness, F is there applied force, and A is the area[1].

The reduced Young's modulus of a tested material is determined using the (2) [6].

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{1}{\sqrt{A}}\frac{1}{C} \quad (2)$$

Where β is the geometrical correction factor for the indenter geometry and C is the compliance [5].

The precision of the test values obtained with (1) and (2) depend on the correct evaluation of the contact area and compliance.

For a Berkovich tip, the projected area of the contact is given by (3) [4].

$$A = 3\sqrt{3}\,h_c^2 \tan^2\theta \quad (3)$$

II. EQUIPMENT AND MATERIALS

The testing was done using the G200 nanoindenter in the SMARTMAT laboratory from the University of Oradea.

In Fig. 2 the nanoindenter used is presented.

Fig. 2. The G200 nanoindenter in the SMARTMAT laboratory.

The tested material is ceramic compound SiO2: 50–65 wt.%. Additional contents are Al_2O_3, K_2O, Na_2O, CaO, P_2O_5, F,Li_2O, ZrO_2, and pigments. Opaquer pastes, Stains, and glazing materials also contain 25–40 wt. % glycols.

In Fig.3 the mounting of the ceramic samples on the motorized stage of the nanoindenteris presented.

For the installation in the motorized stage, a probe holder was used.

Fig. 3.The ceramic sample on the motorized stage of the G200 nanoindenter.

For the testing,a standard Berkovich indenter tip was used. Also, the measurements were realized using the NanoSuite. The NanoSuite is used to run the test with the G200 nanoindenter and can automatically generate histograms and 3D mechanical-properties maps. Graphs and supporting data were exported using this software [7].

III. RESULTS

Test measurements results on the ceramic withgloss coat layer are presented in TABLE I

TABLE I
TEST RESULTS FOR THE FIRST TYPE OF CERAMIC COMPOUND

Modulus At Max Load (GPa)	***Hardness At Max Load (GPa)***	***Drift Correction (nm/s)***	***Disp at Max Load (nm)***	***Max Load (10^{-3}N)***
78,268	6,95	-0,103	2055,483	439,042
78,307	6,896	-0,099	2048,929	434,559
77,12	6,872	-0,106	2050,371	431,448
76,962	6,84	-0,108	2048,692	429,292
77,088	6,835	-0,108	2053,386	431,423
77,955	6,935	-0,108	2052,276	436,415

TABLE I-CONT.

Modulus At Max Load (GPa)	Hardness At Max Load (GPa)	Drift Correction (nm/s)	Disp at Max Load (nm)	Max Load (10^{-3}N)
78,175	6,94	-0,115	2053,103	437,478
76,6	6,796	-0,107	2056,292	430,119
75,717	6,729	-0,12	2051,711	423,791
77,62	6,963	-0,102	2049,671	435,399
76,998	6,868	-0,108	2057,669	434,089
75,087	5,945	-0,089	2050,122	393,885
76,385	6,825	-0,123	2056,187	430,511
76,547	6,769	-0,116	2054,742	428,417
76,401	6,8	-0,098	2046,298	425,618
75,934	6,865	-0,124	2055,171	430,266
76,457	6,881	-0,113	2049,598	429,793
77,877	6,941	-0,117	2054,121	437,192
78,275	6,974	-0,117	2057,614	440,759
78,682	6,934	-0,127	2058,448	440,793
76,923	6,888	-0,121	2050,635	431,622
77,89	6,977	-0,106	2054,859	438,747
77,53	6,888	-0,115	2055,663	435,211
76,401	6,782	-0,111	2048,852	426,051
77,813	7,005	-0,111	2051,246	437,921
76,731	6,769	-0,133	2058,709	430,536
77,403	6,914	-0,12	2058,77	437,105
80,927	7,476	-0,114	2054,269	462,368
76,611	6,746	-0,119	2055,362	428,065
76,238	6,844	-0,121	2052,969	429,413
77,477	6,912	-0,119	2053,988	435,177

The information presented in Table I is obtained using the NanoSuite application. Besides being used to run the test, the application also has the function for generating data analysis reports.

The Information is stored using an common file structure, generally used for an electronic spreadsheet program.

In Fig. 4 the image of ceramic compound whit gloss layer acquired with the G200 CCD camera. On the surface the testing pattern is visible.

Fig. 4. Image of ceramic compound whit gloss layer acquired with the G200 CCD camera.

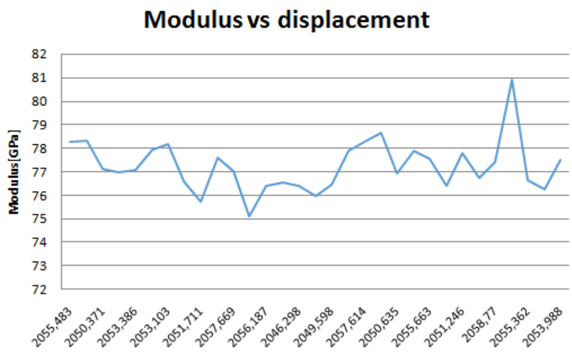

Fig. 5. Modulus of the tested ceramic materials relative to displacement

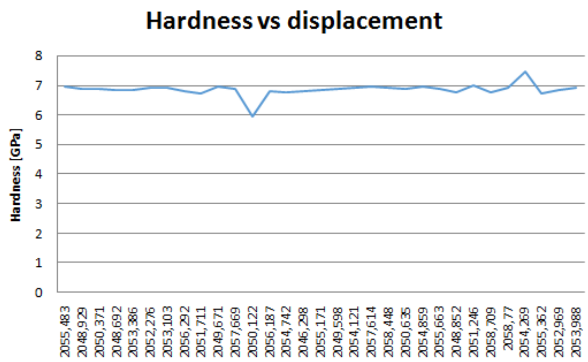

Fig. 6. Modulus of the tested ceramic materials relative to displacement

For the modulus the mean value determined was 77,239 (GPa), for the hardness at maximum load was 6,863 (GPa). The medium displacement in the martial was 2053,394 (nm) and the mean maximum load applied for all tests realized on the material was 432,661(10^{-3}N).

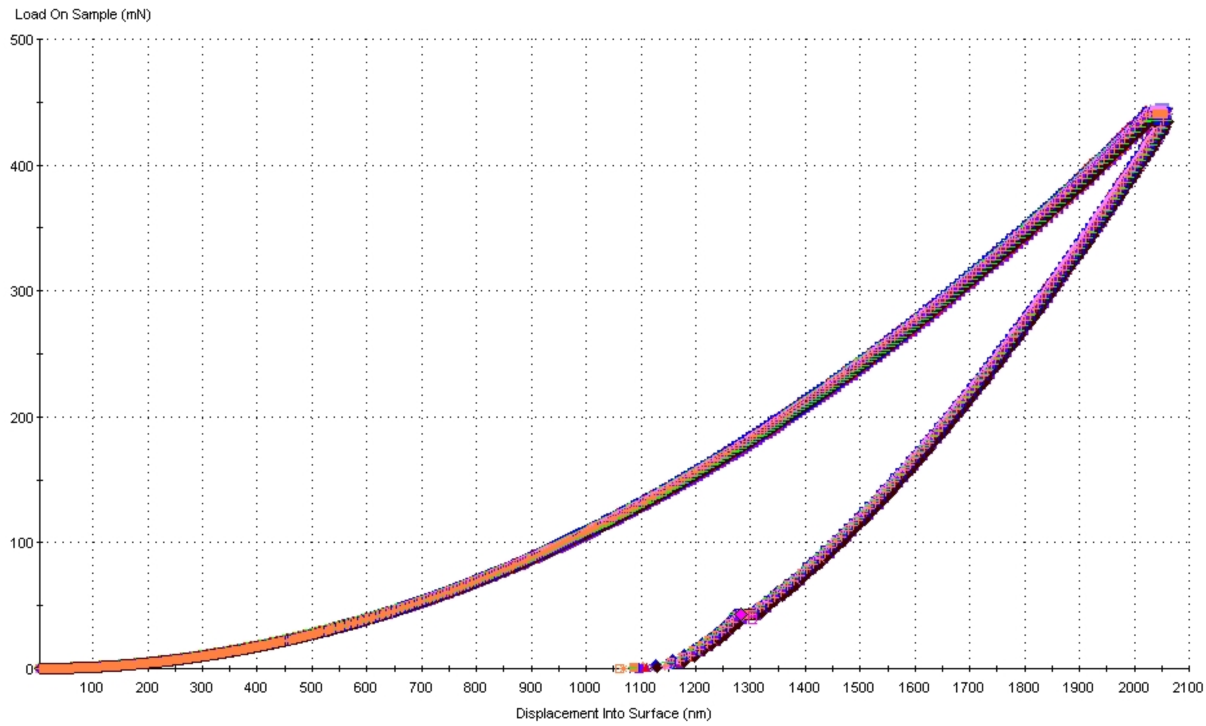

Fig. 7. Displacement of the surface relative to the applied load for ceramic withgloss coat.

Test measurements results on the ceramic without gloss layer are presented in TABLE II

TABLE II

TEST RESULTS FOR THE SECOND TYPE OF CERAMIC COMPOUND

Modulus At Max Load (GPa)	Hardness At Max Load (GPa)	Drift Correction (nm/s)	Disp at Max Load (nm)	Max Load (10^{-3}N)
84,549	5,312	-0,004	2147,024	421,371
83,73	7,654	0,008	2028,625	463,505
76,792	6,694	-0,032	2037,987	419,592
64,637	5,649	-0,008	2035,389	354,442
79,615	6,975	-0,018	2032,7	433,459
37,181	1,342	-0,06	2270,147	140,717
46,737	1,301	-0,051	2120,097	124,986
57,987	2,297	-0,012	2088,151	198,869
80,097	4,881	-0,019	2102,937	376,044
80,826	6,788	-0,042	2074,613	447,753
73,578	6,776	-0,043	2042,631	416,229
73,556	6,603	-0,045	2043,73	411,057
74,12	6,419	-0,051	2040,02	404,796
69,692	5,64	-0,048	2042,604	368,193
66,035	5,289	-0,038	2040,886	346,69
66,196	3,703	-0,037	2045,673	278,969
77,693	5,759	-0,017	2053,848	393,326
72,138	4,962	-0,035	2046,888	347,356
82,316	7,506	-0,033	2031,656	456,706

Modulus At Max Load (GPa)	Hardness At Max Load (GPa)	Drift Correction (nm/s)	Disp at Max Load (nm)	Max Load (10^{-3}N)
76,925	6,804	-0,034	2033,867	421,856
76,955	6,557	-0,029	2033,685	413,55
73,521	6,846	-0,015	2028,969	412,745
66,938	5,572	-0,019	2030,556	355,428
75,305	6,881	-0,066	2043,706	424,423
80,947	7,226	-0,091	2048,335	451,724
85,092	6,198	0,012	2024,918	413,388

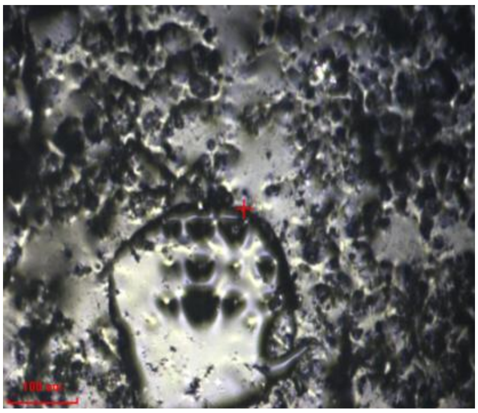

Fig. 8. Image of ceramic compound without gloss layer acquired with the G200 CCD camera

In Fig.8 the image of ceramic compound without gloss layer acquired with the G200 CCD camera.

On the surface the testing pattern is not as easy to distinguish as in the test sample presented in Fig.4 (the ceramic with gloss coating).

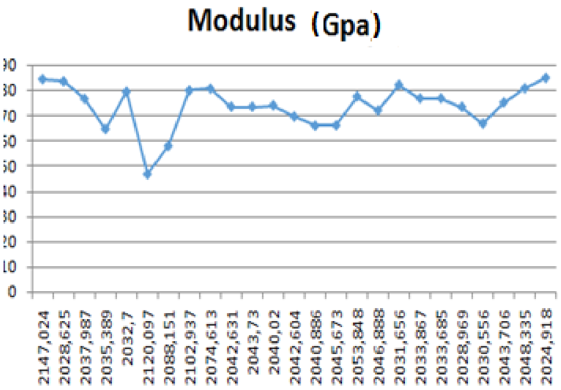

Fig. 9. Modulus of the tested ceramic materials relative to displacement

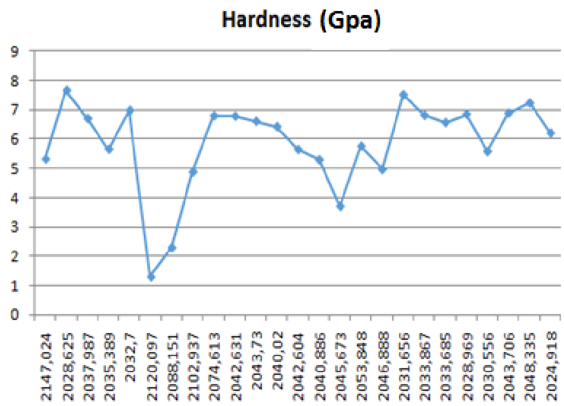

Fig. 10. Hardness of the tested ceramic materials relative to displacement

For the modulus the mean value determined was 72,429 (GPa), for the hardness at maximum load was 5.678 (GPa). The medium displacement in the martial was 2060,371 [nm] and the mean maximum load applied for all tests realized on the material was 372,968 (10^{-3}N).

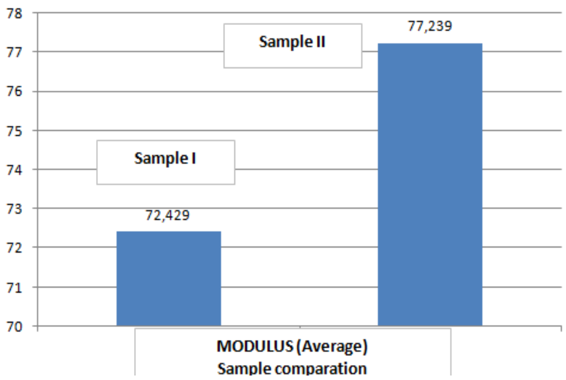

Fig. 11. Comparison of modulus average value between samples (with and whiteout glazing)

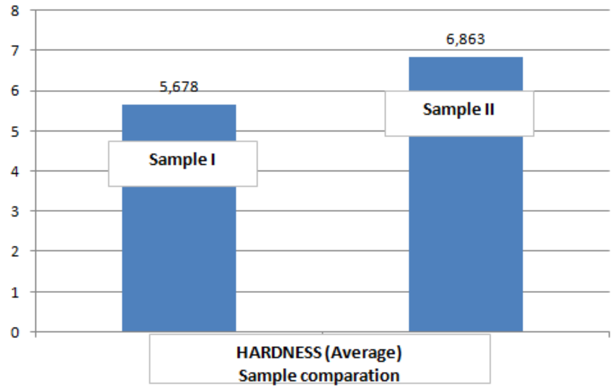

Fig. 12.Comparison of hardness average value between samples (with and whiteout glazing)

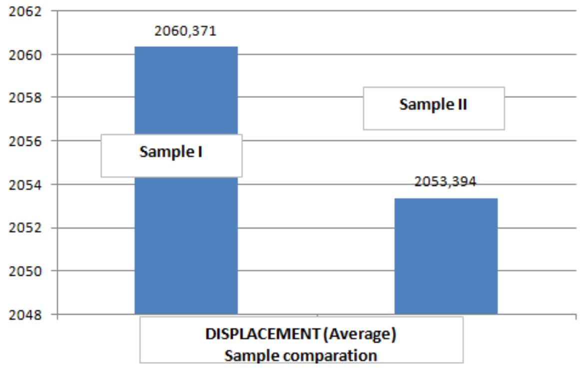

Fig. 13 Comparison of displacement average value between samples (with and whiteout glazing)

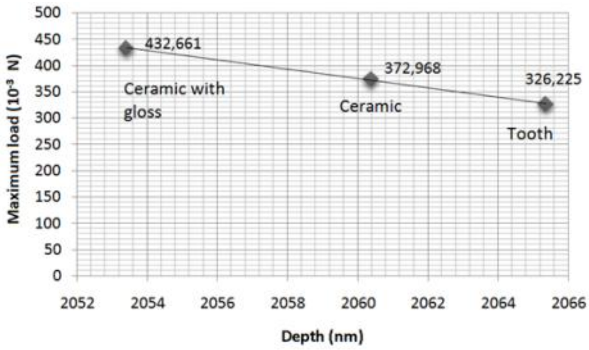

Fig. 14 The comparison of displacemtn vs load for ceramic with gloss, simple ceramic and the values for a tooth

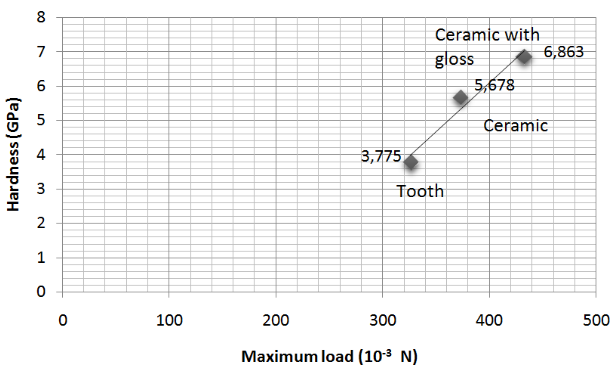

Fig. 15 The comparison of displacement vs harhness for ceramic with gloss, simple ceramic and the values for a tooth

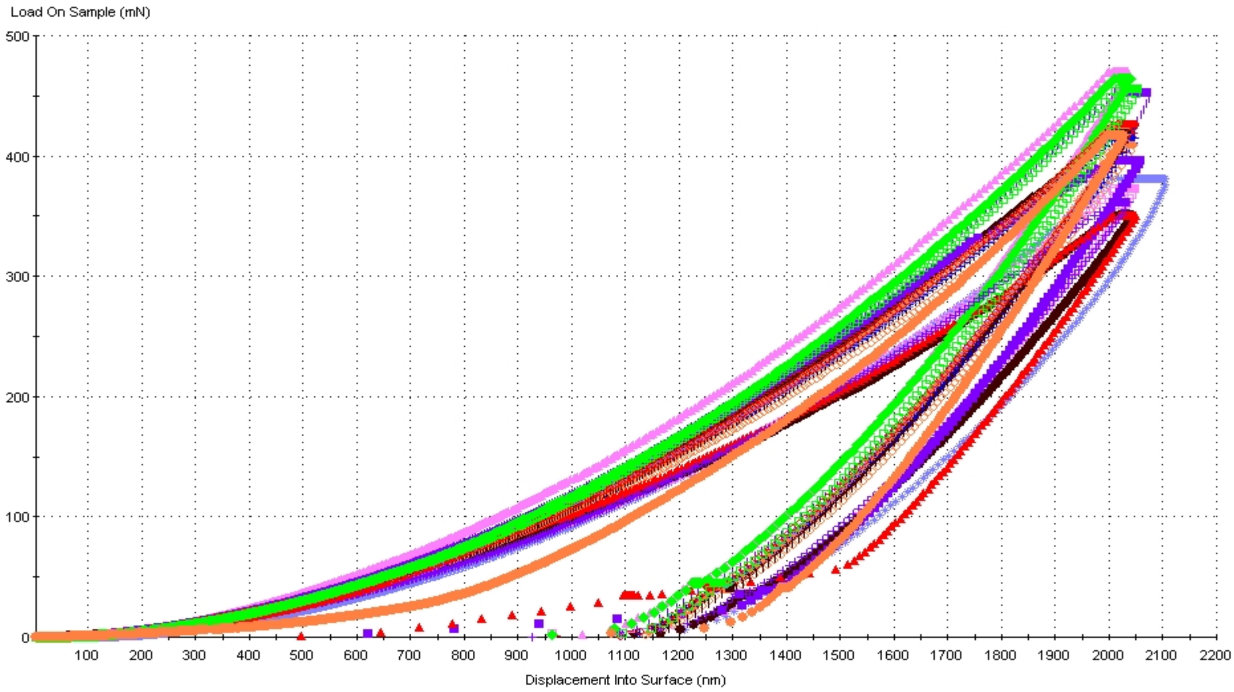

Fig. 16. Displacement into the surface relative to the applied load for ceramic without gloss layer

IV. Conclusion

This method of characterization is not only perfect for very hard types of materials like the ceramics we tested SiO2: 50–65 wt.% Al_2O_3, K_2O, Na_2O, CaO, P_2O_5, F,Li_2O, ZrO_2, but also for multi layer smart materials like SiO2: 50–65 wt.%, glycols 25–40 wt. % Al_2O_3, K_2O, Na_2O, CaO, P_2O_5, F,Li_2O, ZrO_2 which present different behaviors at different depths.

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