# COMPARISON OF THE NANOINDENTATION RESULTS OF TWO GENERATION OF POWDER METALURGY PRODUCED MATERIALS FOR PLASTIC INDUSTRY

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***Abstract:***

*This article analyses the properties and nanoindentation characteristics of the stainless PM steels M390 Microclean® and M398 Microcelan®. In a first part, the literature sources on PM steels, stainless steels and thermochemical treatment were studied in order to collect information on the problems that can be found in the thermal or thermochemical treatment of M390 and M398 to improve their mechanical and anticorrosion properties. Optical microscopy of both materials is presented to show the structure and get a more complex picture about them. The main reason for this article – the nanoindentation was performed on the Hysitron Triboindenter TI 950. For both samples, 7 indentation points were selected on the SPM scan – 4 for carbides and 3 for the base matrix. Based on the results, the nanohardness H and the reduced modulus of elasticity Er as well as the reduced modulus of elasticity of the phases ES were determined. The nanohardness H of the matrix corresponds to that of the ferrite, which confirms the material manufacturer's specifications. The nanohardness of the carbides corresponds to the results of the other materials tested by CEDITEK.*

## 1 Introduction

Metallic materials used in the processing of plastics on injection moulding machines or in injection moulds are often subjected to mechanical stress, high temperatures, high pressure, abrasion or chemical influences. For this reason, manufacturers of metal materials have worked together with equipment manufacturers in the plastics industry to develop materials that can withstand all these conditions. For most applications, metallic materials are used that are produced using the powder metallurgy method HIP (Hot Isostatic Pressing). These materials are mainly used for the components of injection moulding screws and injection moulds. These materials also have excellent properties for the food industry, where they have a wide range of applications. [1] The materials produced by powder metallurgy are particularly interesting in terms of their composition. This technology makes it possible to produce alloys that cannot be manufactured using conventional metallurgical processes, as can be clearly seen in the materials analysed by Boehler, manufacturer of the materials M390 Microclean® and M398 Microclean®. Research on powder metallurgical steels is still rare in the scientific literature and there are not many authors dealing with this topic. For this reason, the literature search was focused on Powder Metallurgy Steels but also on anti-corrosion steels with good abrasion properties and their properties after nitriding, thermal and thermochemical processes, which are analysed in the following articles. I. Braceras Et Al. [2] treated anticorrosion steel by high-density plasma nitridation. One of their findings was that corrosion resistant steels, especially on martensitic ones after the nitridation increased surface hardness, but the corrosion resistance decreased. According to their investigation the reason of corrosion resistance decreasing is the chromium, especially chromium nitrides occurring during the nitridation. On the

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steel 1.4545 (15-5PH) Braceras Et Al. tried high density plasma nitridation. Following tests discovered that thanks to high density plasma nitridation the uniform surface nitridation layer was made without cracklings which resists the insertion of corrosion inhibitors to the material under the nitridation layer.

It increased the anticorrosion properties of the material. Authors G. H. Farrahi and H. Ghadbeigi [3] analysed influence of different kinds of surface treatments for the increasing of fatigue life of AISI D3 steel. During the tests they found out that nitridation and carbonitridation on one hand increased abrasion resistance, but on the other hand decreased the fatigue life and resistance against the cracklings because the high surface hardness in comparison with the basic material caused initiation of fatigue cracklings. One of the reasons was increased surface roughness of material after the nitridation because the microstructure homogeneity changed the material surface and it increased probability of the crackling occurrence. Authors found out also that after the nitridation or carbonitridation there originated the porous structure. M. Černý Et Al. [4] proceeded Rapid Solidification Powder (RSP) steels. One of the steels was Böhler M390. The output of the article is that minimal dimensions of the grains and rounded carbides shapes in powders steel expressively increases value of critical stress conditions. Authors estimate that the toughness of M390 steel is mostly caused by the absence of microstructural cracklings. On the edge of the grains concentration of the stress is not present because according to standard models very fine carbides do not provide conditions for formation of micro cracklings nucleation in martensitic grains. A. Blutmager Et Al. [5] designed apparatus for testing of materials used for plastification units and its components.

They proposed complex methodology of testing which has not existed before. Tests were performed with the steel produced by powder metallurgy (X190CrVMo20 4 PM) which corresponds to a M390 steel and the same steel but with the CrN surface treatment. For the tests the plastic stick made of glass fibres filled material Polyamid 66-GF50, Ultramid A3WG10 from producer, company BASF was prepared. After the process overrun at defined parameters – pressure, rotations (metal sample had a circular shape and rotated) they investigated the surface by roughness measuring device and a microscope. They found out that higher abrasion resistance had material with the CrN coating than material without coating, even that the surface abrasion was low in both cases. L. Jinglong Et Al. [6] in the article analysed electrochemical nitridation on the corrosion resistant coating created on the duplex stainless steel 2205. During the tests they found out that nitridation at lower temperatures effectively slowed down massive precipitation and allowed to form super stable Nitrogen austenitic phase. Results of this work showed even that the surface layer had reduced Chromium volume by the creation of chrome nitrides the corrosion resistance increased due to a stable and compact passive deposit which arose during the electrochemical nitridation. Team of C. J. Scheuer [7] was focused on carburizing of martensitic stainles steel AISI 420. From the scope of corrosion resistance they found out coherence between carburizing time and corrosion resistance. From their results ensues that the corrosion resistance increased in comparison with the material in base state but the longer carburizing process run the lower corrosion resistance was, especially due to a crome carbides creation. It implies that for saving the corrosion resistance it is necessary to keep the temperature at lowest possible value to avoid occurrance of unwanted compositions of several components. In their article A. Zangiabadi Et Al. [8] described creation of martensitic austenite during the nitridation of martensitic duplex steel 2205. Final findings showed that high concentration of Nitrogen interstitials cannot be created in martensite or ferrite even not during the nitridation by the reason of creating martensitic austenite. During the tests they nitrided the steel at low temperatures to get massive supersaturation what proved in a transformation of martensitic martensite-austenitic phase transformation or into the martensitic ferrite-austenitic transformation. Group of authors under the leadership of L. Marot [9] was concerned with the increasing of nitridation efficiency and increasment of mechanical properites of staniless steels mainly at low temperatures under 550°C.

Their research showed that during the nitridation in $N_2$-$H_2$ plasma it is possible to achieve 10 μm depth at temperature 430 °C while with conventional methods effect of nitridation is decreased at temperatures lower than 550 °C. During their tests with stainless steel 304L after nitridation the Young´s modulus remained without the change and surface hardness increased three times. The results imply the range of suitable temperatures where stainless steels must be nitrided to avoid creation of chrome nitrides and by that to decrease anticorrosion protection of material. In their next article, A. Blutmager et al [10] investigated the erosion behaviour of plastics, in particular of polyamide with 50 % glass fibres, for three different steel alloys. One of the materials was powder metallurgically produced steel (according to the content it was Boehler M390). Using an electron scanning microscope, the authors found that the erosion process mainly took place on the base material. The carbides were almost intact and remained almost in position in the matrix without any serious signs of wear. During the testing of “Zero time holding quenching“ A. Li [11] achieved intresting results by the special principle of quenching when there is no holding time for the overheating of whole cross section of material, but the cooling down comes directly after reaching of asked temperature on the surface. This proces helps decrease the oxidation, decarburizing and other problems which occure during the holding time. Zero time holding quenching is able to decrease the deformation of the parts in wide range during the quenching. According to A. Li “The austenite inverse transformation during zero time holding quenching at lower temperature can notably improve the mechanical properties of 27SiMn steel“. This method should be interesting in the future for testing on the Powder Metalurgy Steels Böhler M390 and M398.

The authors J. Procházka, Z. Pokorný and D. Dobrocký [12] from the University of Defence in Brno have solved the nitriding layers for materials used in the defence industry in their article. Although the applications are somewhat different from the PMS used in the plastics industry, the article contains very interesting information. The most interesting information seems to be the following: “Due to the great similarity of the atomic radii of carbon (71 pm) and nitrogen (65 pm), these interstitial elements can occupy the same positions in a crystal lattice. It can therefore be assumed that a reduction in the carbon content in a steel could lead to an increase in the diffusivity of nitrogen.“ This information should be interesting for the comparison of the nitride layers of the materials M390 and M398, as M398 has a significantly higher content of carbon, which is mainly bound in carbides, but should have a different influence on the chromium precipitation and also on the rising of the nitride layer in the comparison. In view of the chromium and its diffusion with nitrogen, this fact may mean that the carbides do not decompose during nitriding and subsequently diffuse with nitrogen, but that the chromium carbides remain stable. This information will be the subject of further investigations.

## 2 Experimental investigation

### *2.1 Basic information*

Materials Böhler M390 Microclean® and M398 Microclean® are martensitic chromium steels which both have high range of alloy components, mainly Chrome. These steel alloys regarding their content do not have equivalent in the standards ISO, EN and others, but it is possible to assign them to group of highest quality high speed steels. Their biggest advantage is high corrosion and abrasive resistance mainly influenced by high content of Chrome and other components, but also a technology of their production – HIP (Hot Isostatic Pressing). For its properties the steels are used mainly for processing of plastic materials with abrasive fillers as a glass fibres and also materials with aggressive chemical components as Chlorine (PVC) or Sulphur (PES). All these corrosion and abrasion factors affect the material of the screw during the hard conditions of high temperature (common processing conditions of plastic materials are in the range from 220 °C to 350 °C, exceptionally up to 450 °C) places high demands on screw and barrel. Material of plasticizing unit must resist these conditions on a long-term basis to avoid damage of the functional parts of injection molding machines and tools. [15]

### *2.2 Chemical content and mechanical properties of materials*

Both materials are produced by HIP (High Isostatic Pressing) as mentioned before, where the liquid material is atomized by inert gas. Very fine spherical shaped powder which was produced by atomization is filled into the special container. In the container the vacuum is created to get gas from the space between powder grains. Gas can cause material defects. Then the container is closed and put into the furnace where the material is slagged under the high pressure (usually used pressure is from 40 up to 160 MPa). Usual pressing temperature is from 1150 up to 1200 °C (0.8 * solidus temperature). [16] The advantage of these powder metals production technology is that thanks to removing of the gas and high pressure during the process material is not porrous what ensures its excelent mechanical properties. By the temperature lower than solidus the powder grains are not fully melted and the junction of material grains is provided by melted surface of powder grains. It helps to keep the grains of several components nearly in original position as it was in the powder form. Both materials contain high volume of alloy components which are occurred in the matrix as carbides. These components helps material to get excellent results which predestinates it for use in very hard conditions. Nowadays powder metalurgy steel M390 is used for applicatons in plastic industry especially for its uniqe anticorrosion and antiabrassive properities. It is mainly used for production of injection molding machines screws which are intensively stressed by high abrrasion, heat, chemical and mechanical factors. [17]

M398 PMS steel is new generation from the same producer, company Böhler. According to available information it has better resistance against wear and slightly lower but still comparable resistance against chemical influences. This material will not replace M390, but they will coexist and customers would choose which application is suitable for them.

*Table 1. Chemical content and mechanical properties of M390 Microcelan® [4]*

| *Chemical content and mechanical properties of M390 Microcelan®* | | | | | | | |
|---|---|---|---|---|---|---|---|
| Component | C | Si | Mn | Cr | Mo | V | W |
| Content % | 1.9 | 0.7 | 0.3 | 20.0 | 1.0 | 4.0 | 0.6 |
| Density at 20 °C | | | | 7.54 kg/dm$^3$ | | | |
| Thermal conductivity at 20 °C | | | | 16.5 W/(m.K)f | | | |
| Hardness – supplied condition | | | | 280 HB | | | |

*Table 2. Chemical content and mechanical properties of M398 Microcelan® [1]*

| *Chemical content and mechanical properties of M398 Microcelan®* | | | | | | | |
|---|---|---|---|---|---|---|---|
| Component | C | Si | Mn | Cr | Mo | V | W |
| Content % | 2.7 | 0.5 | 0.5 | 20.0 | 1.0 | 7.2 | 0.7 |
| Density at 20 °C | | | | 7.46 kg/dm$^3$ | | | |
| Thermal conductivity at 20 °C | | | | 15.2 W/(m.K) | | | |
| Hardness – supplied condition | | | | 330 HB | | | |

### *2.3 Analysis of microstructure*

As part of the material investigation, the decision was made to carry out a light microscope test. The reason for this was simple – to find out what the microstructure looks like and whether the different content of components has an effect on the different microstructures. The NEOPHOT 32 optical microscope with the Canon digital camera was used for the investigation. The photos from the microscope were sent directly to a connected PC. The samples were etched with a mixture of acid and glycerine: 10 ml HNO3 + 20 ml HCl + 20 ml glycerine + 10 ml H2O2. The initial etching with Nital confirmed the extraordinary corrosion resistance of both materials, as the visualisation of the structure was not sufficient. The samples were etched in the glycerine mixture for 10 seconds, then cleaned with distilled water and alcohol and dried with hot air.

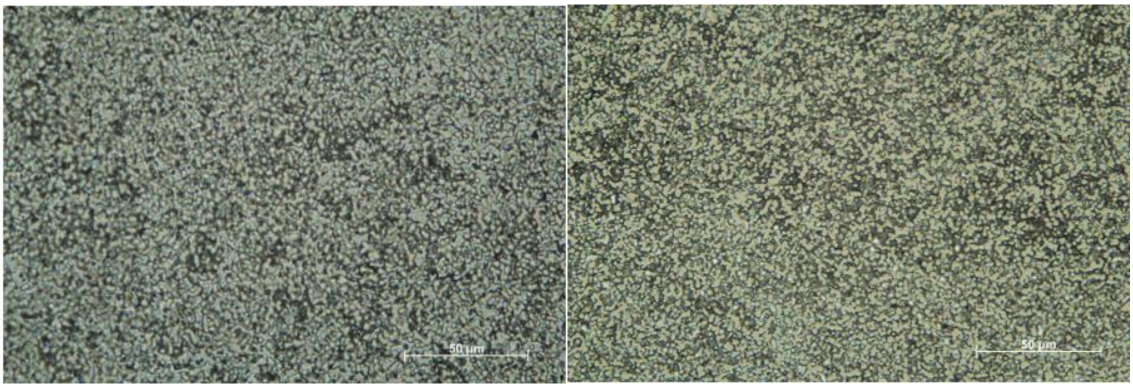

*Figure 1. Structure of material M390 (Left) and M398 (Right)*

Based on photographs, both materials appear to be visually similar. M398 has a higher proportion of alloy components than M390 (32.6 %: 28.5 %) and it can be assumed that the proportion of light-coloured fields is greater in M398 than in M390. The darker patches are the base material (ferite), the lighter patches are alloy components, mainly carbides. The visual aspect confirms the information provided by the material manufacturer, according to which M398 contains a higher proportion of additional components than M390. At the same time, it is possible to see how fine the structure of the material is. At 1000x magnification, it is not possible to recognise more than light and dark spots. For this reason, the materials must be analysed using a scanning electron microscope (SEM).

### 2.4 Experimental methods

Quasistatic nanoindentation – testing of the material hardness in range of nanometres ($m^{-9}$) is possible by pressing the tip of indenter with defined geometry (Vickers – square pyramid with apex angle 136°, Berkovich – apex angle of walls 65° 27´, Knoop – quadrilateral pyramid indenter with two different face angles 130°and 172°30´, Cube-Corner indenter – face angle 35°26) determines hardness except on the level of several structural components of material, what in case of alloys is produced by powder metallurgy desirable. During the nanoindentation in comparison with standard hardness measuring methods it is not possible due to a size of indenter tip to estimate exact dimension of the imprint. Because of this the main measuring parameter is the depth of the indenter penetration into the samples surface what together with the known shape of indenter allows to estimate the contact surface indirectly. This method can be used for achieving values of the hardness as well as local Young´s modulus of elasticity. [13]

These values are realized from the nanoindentation curve. Its interpretation was examined by many authors. Most used method of its evaluation is "Oliver – Phaar", which determines already mentioned Young´s modulus of elasticity *Er* and nanohardness *H*. During the nanoindentation of powder metallurgy produced materials it will be interesting to measure hardness of carbides in basic matrix with following comparison of these results within the scope of one material, but also mutual comparison of both materials. Hysitron Triboindenter TI 950 apparatus was chosen for nanoindentation. As a part of nanoindentor there is evaluation software Triboscan, which processes the results of nanoindentation. Device is a part of CEDITEK laboratory (Center for tesing and diagnosis of materials) as a part of research infrastructure at Faculty of Special Technology in Trenčín. Measurements was provided by indentor with Berkovich geometry (apex angle of walls 65° 27´).

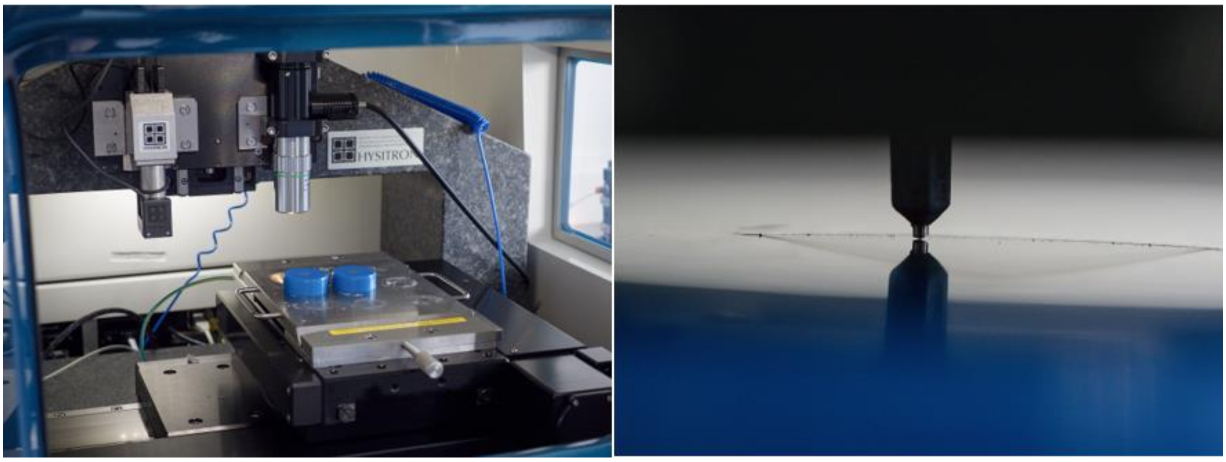

*Figure 2. Nanoindentor configuration and indentation tip during the performed tests of material*

Before start-up of measurements the samples, same as used for optical microscopy, had to be fixed to the surface of movable micromovements support of nanoindentor. After the fixation it was necessary to set the position of indentation by accelerated movement of the support. After the definition of exact positions of indentation for each sample – 7 positions for both samples (0-6) the process of indentation was started consequently. The apparatus realized the process independently according to pre-set indentation points.

Nanoindentation parameters:
Dimensions of indentation field: 20 x 20 µm,
Impress regime: trapezoid
Maximum load: F = 8000 µN
Indentation time: $t = 2$ s

After the finishing of the quasistatic nanoindentation process the apparatus sent the data for evaluation to connected computer with evaluation software Triboscan where whole surface SPM scan (Scanning Probe Microscopy) was processed as well as nanoindentation curves (Load – Displacement *P –h)* and final results of nanohardness *H* and reduced Young´s modulus of elasticity *Er*.

## 3 Results and discussion

The penetration points were chosen so that some of them were located on the tips of possible carbides (0-3) and the rest between these grains (4-6), at the points where the base material, which according to the manufacturer should be ferrite, was most likely to be found. The manufacturer supplies both steels in the annealed condition, in which the steel does not yet contain martensite but does contain ferrite. Martensite is formed after heat treatment for maximum wear resistance or maximum resistance to chemical erosion.

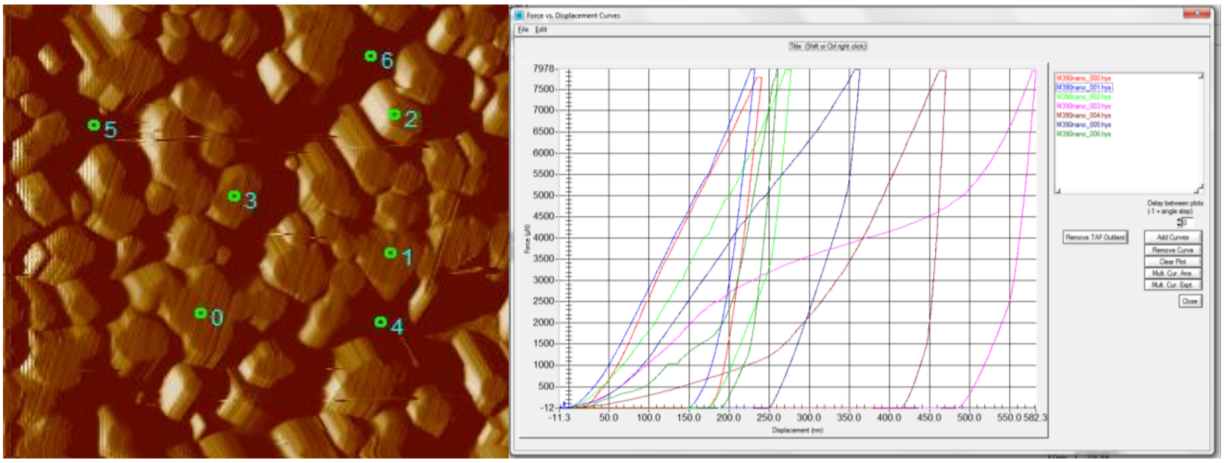

*Figure 3. SPM Scan of M390 with placement of particular indentation positions and nanoindentation curves*

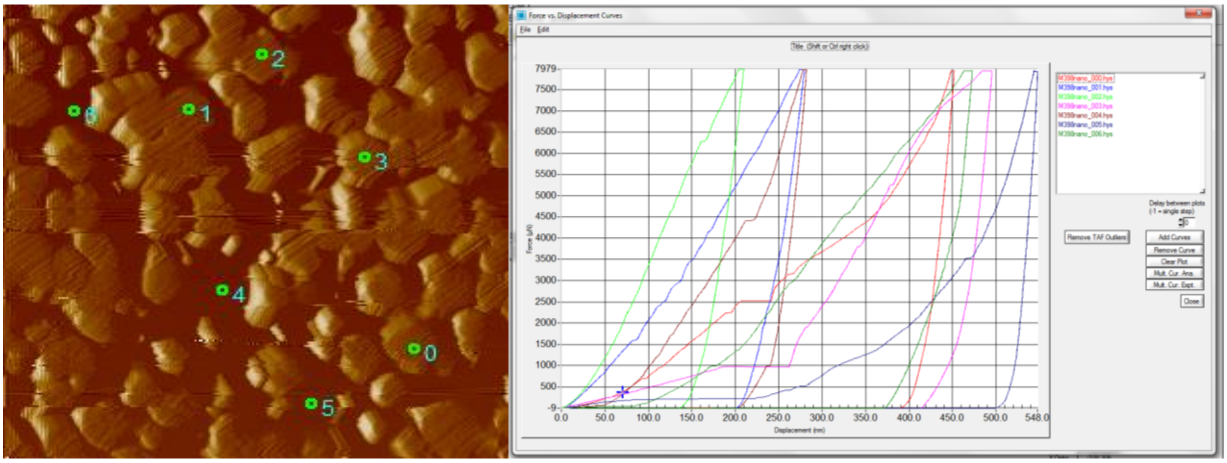

*Figure 4. SPM Scan of M398 with placement of particular indentation positions and nanoindentation curves*

From realised measurement results that in both tested materials were in chosen points 0-3 measured higher values of nanohardness *H*, also the reduced Young´s modulus *Er* was higher. The theoretical assumption that the points 0-3 are carbides and points 4-6 are basic matrix of examined steels - Ferite was most likely confirmed.

*Table 3. Measured local nanomechanical properties components structure of tested M390 Microcelan*

| Position | Reduced Young´s modulus - reduced modulus of material elasticity *Er*(GPa) | Nanohardness H(GPa) | Structural component |
|---|---|---|---|
| 0 | 160.22 | 7.26 | Carbide |
| 1 | 174.26 | 8.06 | Carbide |
| 2 | 160.20 | 5.40 | Carbide |
| 3 | 162.07 | 5.04 | Carbide |
| 4 | 124.58 | 2.56 | Ferite |
| 5 | 116.91 | 3.16 | Ferite |
| 6 | 123.53 | 3.74 | Ferite |

*Table 4. Measured local nanomechanical properties components structure of tested M398 Microcelan*

| Position | Reduced Young´s modulus - reduced modulus of material elasticity *Er*(GPa) | Nanohardness H(GPa) | Structural component |
|---|---|---|---|
| 0 | 163.15 | 5.75 | Carbide |
| 1 | 165.62 | 6.29 | Carbide |
| 2 | 174.54 | 7.16 | Carbide |
| 3 | 157.66 | 5.45 | Carbide |
| 4 | 127.54 | 2.88 | Ferite |
| 5 | 117.77 | 2.15 | Ferite |
| 6 | 116.39 | 2.66 | Ferite |

Calculation of Young´s modulus of elasticity of phases $E_S$ [GPa] for examined materials was calculated from equation (1):

$$E_s = \frac{(1 - v_s^2)}{(\frac{1}{E_r} - \frac{1 - v_i^2}{E_i})} \tag{1}$$

$E_i$ – modulus of indentor, $v_s$ – Poisson´s constant of the sample, $v_i$ – Poisson´s constant for the indenter Berkovich type, $E_i = 1141$ GPa, $v_i = 0.07$ a $v_s = 0.285$.

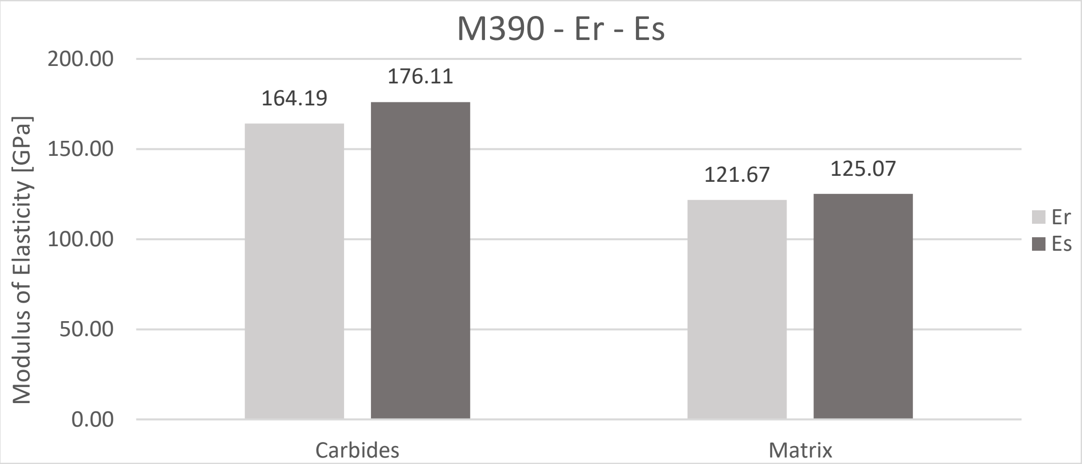

*Figure 5. Comparison of measured Young´s modulus Er and calculated modulus of phase Es for M390*

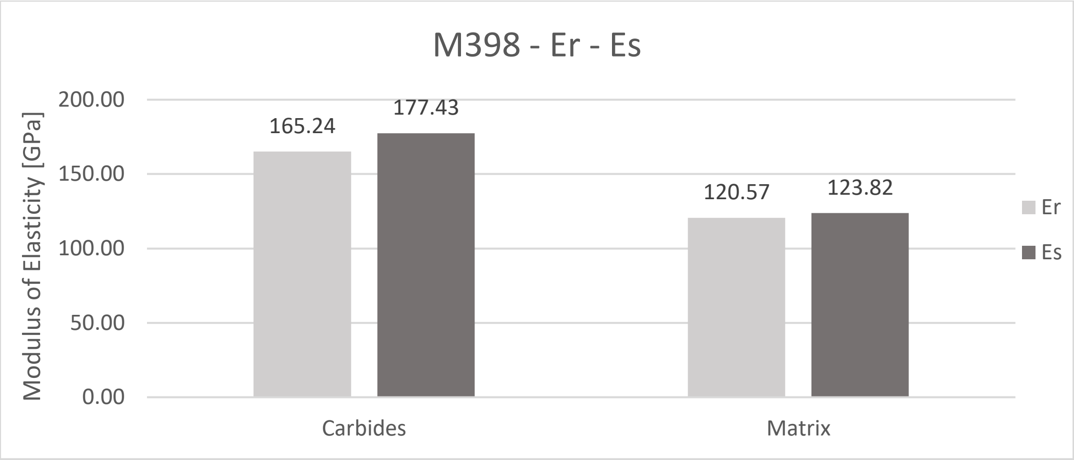

*Figure 6. Comparison of measured Young´s modulus Er and calculated modulus of phase Es for M398*

With regard to the minimal differences between the hardness of the individual components (carbides and matrix) of the two materials, we can assume (also based on the article by A. Blutmager Et Al. [10] – the carbides did not fall off during the test and remained in their positions in the material – the abrasion mainly affected the base matrix of the material) that the higher abrasion resistance of the M398 material compared to M390 is the result of the larger volume of the alloy components (32,6 % in M398 and 28.5 % in M390), which hinder the erosion of the base material in such a way that the larger surface area of the carbides does not allow the abrasive particles to penetrate the base matrix and remove their particles. An important result arises from the nanoindentation - base material is really Ferrite. It was found out from the comparison with other results of CEDITEK scientific workplace that assumed material has the same hardness as provable Ferrite in other materials.

## 4 Conclusion

The nanoindentation of the PM steels M390 and M398 is a first phase of the project to improve their performance properties. It has helped to increase the knowledge of these two materials, but it is not sufficient to fully understand their properties. Nanoindentation together with optical microscopy is sufficient as a basis for the following tests. Thank you to these two tests, it is possible to recognise the very fine structure of these PMS steels and to point out that they do not have the appearance and structure of standard manufactured steels. At 1000x magnification, it is not possible to see normal grain boundaries, distinguish different phases or define other material components. It is necessary to examine the material in more detail using equipment with higher magnification and screen resolution. It would be possible to start from the manufacturer´s catalogue images, but in this case it is necessary to verify them and find out their correspondence with the real appearance of the material. By nanoindentation and comparison of the values obtained with other known materials, it was possible to prove that the basic matrix of both materials as supplied is ferrite. The manufacturer presents the material as martensitic steel, but this state of the material becomes the required state (highest abrasion resistance or highest corrosion resistance) after heat treatment. Further tests are necessary to get to know the material better, especially the examination under the scanning electron microscope (SEM), but also other tests and the repetition of nanoindentation after heat treatment. These tests will provide a consistent overall picture of the materials and their properties after thermal and thermochemical treatment.

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