

## Comparative characteristics of endodontic drills

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The work concerns the analysis of influence of the wear process of endodontic instruments on the mechanical and physico-chemical properties of the materials from which they are made. A detailed study of the microstructure, mechanical properties and corrosion resistance in the environment simulating work of the tool was conducted. The research was done for the new Mtwo endodontic files and after six times of use. In addition, the observations with a scanning electron microscope in order to reveal possible damage caused by the impact of a corrosive environment were carried out. The results showed that use of the tool by six times revealed damage at the edges of the blades and may cause a lack of continuity of the cutting line resulting in the uneven distribution of the resistive force which acts on the tool during operation.

*Key words:* *endodontic instruments, Ni-Ti rotary instruments, nickel-titanium alloys, Mtwo files*

### 1. Introduction

In modern dentistry Ni-Ti alloy is commonly used for producing the instruments for the root canal preparation due to its many advantages [4], [12], [25]. Definitely greater susceptibility to deformation of the tools made of superelastic Ni-Ti alloy, compared to a martensitic steel, allows reduction in the incidence of complications during the preparation of curved root canals. Tools made of Ni-Ti alloy ensure the preservation of the natural shape of the canal as well as its effective extension at the top part, which is important for the success in root canal treatment [4], [7]. Furthermore, tools made of Ni-Ti alloy are more resistant to wear than their counterparts made from stainless steel [19]. In addition to the many advantages of nickel-titanium tools, they have also several disadvantages compared to traditional tools made of stainless steel. The main defect of tools produced from Ni-Ti alloy is low surface hardness, causing worse cut-

ting properties, and also higher tendency to breaks at the top of endodontic files. A complex manner of producing the nickel-titanium tools should be included to the disadvantages [7], [12], [23]. The production of triangular or square cross-section tools and further twisting them, as is in the case of steel tools, is not possible. The shape of the tool must be formed from bulk material, by interrupting the continuity of the structure of the metal and the creation of grooves inclined to the axis of the tool which increases the susceptibility to fracture [12]. Eggert et al. [7] have studied the structure of LightSpeed tool under the scanning microscope and have found that the damage of working edge in the form of pitting was visible on the tools already after use, as well as in the new ones. Imperfections of the new tools result from the rapid blunting of the machines during the processing of nickel-titanium alloy. The observed damage of the outer surface does not affect the stability of the instruments in the root canal [7]. Despite a significant flexibility, nickel-titanium instruments are subject to

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fracture, when the stress in the tool exceeds its strength [14], [23]. The main cause of fracture results from low fatigue strength of the material [8]. The defect of Ni-Ti instruments reduces their bending resistance by about 25–50%, and increases the susceptibility to fracture as compared to the instruments of the square cross section made of stainless steel (K-Files) [7]. Unfortunately, the degree of wear of the tools made of Ni-Ti alloy is not visible macroscopically [9]. Only the observations in the scanning electron microscope allow the differences in the degree of wear to be identified [12]. The blades of rotary tools made of Ni-Ti alloy become quickly dull and therefore require frequent replacement depending on the number of channels preparation, the size of tools and anatomical structure of root canal [7]. According to the manufacturer recommendations, the number of treatments with the use of nickel-titanium instruments is usually 4–16 times on average. If a file shows any change of the shape the manufacturers do not recommend its use [8], [9]. Research has shown that the rotary nickel-titanium instruments shear root dentin less effective than the rotational tool made of steel [21], [24]. Previous quality criteria for the materials used for tools do not specify recommendations for such a miniaturized geometry of endodontic instruments. Presently, work on the methods of surface modification of the files made of Ni-Ti alloy is conducted. The tests include the use of CVD techniques to improve the micro-hardness, resistance to corrosion and wear, as well cutting efficiency [18], [20]. Currently, there are no comprehensive studies on the impact of technological and surface treatments on the abrasion and corrosion resistance as well their changes as a result of sterilization process. The studies show influence of the conditions of the use of Mtwo endodontic files made of Ni-Ti alloy, including the sterilization process, on the mechanical properties, microstructure, corrosion resistance and changes in the quality of the cutting surface [2], [3]. Clarification of these issues will be beneficial to further optimizing the properties of selected tools, including endodontic files.

## 2. Material and methods

The Mtwo endodontic files for root canal therapy, made of Ni-Ti alloy with the chemical elements concentration of 56% Ni and 44% Ti were used in the research. The new file and those used six times were analysed in the study. Mtwo system is more often used to root canal therapy. Treatment of the canal is carried

out smoothly along the entire length, shaping the canal up to its top, with the use of the tools from the smallest to the largest. The size of endodontic files are marked with the appropriate colour of the handle portion. Taper shape of the tools can also be easily determined by the number of cuts (fringes) on the handle (taper 0.04 – 1 band, taper 0.05 – 2 bands, taper 0.06 – 3 bands, taper 0.07 – 4 bands). Two groups of the files with different sizes were used in the research (Fig. 1). Endodontic files marked as white are used as second in order for the root canal treatment. They have dimensions 15/05, where 15 is the top diameter of 0.15 mm, and 05 determines the taper shape (increasing the diameter in the direction of the handle) of 0.05 mm (5%) per millimetre of length. Files marked as yellow have dimensions 20/06 and are used just after the white ones. All files have the same length of 16 mm for the working part. New files were delivered in a sterile state. Steam sterilization was conducted for 12 minutes in an autoclave Basic Plus by Powers, at 134 °C and at a pressure of 2.1 bar. Sterilization of dental files was carried out in accordance with the requirements of PN EN 556-1:2002 [15].



Fig. 1. Mtwo endodontic files used in the research

Scope of the research included the metallographic microscopic studies, hardness measurements, corrosion resistance tests and observations in the scanning electron microscope. The metallographic microscopic study was carried out to identify the structure of material used for endodontic Mtwo instruments and to evaluate the non-metallic inclusions level. Measurement of grain size was carried out according to the recommendations of ASTM F 2063-00 [1].

Observations were performed using the light microscope LEICA MEF4A at 100 to 500 $\times$  magnification. Longitudinal and transverse specimens made of new Mtwo endodontic files and those after use were applied in the metallographic microscopic research. Hardness measurements were made by the Vickers hardness test method. Tests were conducted on the longitudinal and transverse metallographic specimens. Hardness of the working parts of files was measured with load of 0.409 N. Tests were performed on the basis of the PN-EN ISO 6507-1:2007 and PN-EN ISO 4516: 2004 standards [17] and Zwick ZHR micro-tester with software FM-ARS 9000 was used.

Determination of resistance to pitting corrosion was conducted using the PGP 201 potentiostat radiometer, which allows the value of the electrode potential to be changed according to the established rate. The tests were performed on the basis of PN-EN ISO 10993-15:2005 and PN-EN ISO 17475:2010 standards [16].

The study of corrosion resistance of biomaterials and medical devices is carried out in terms reflecting the conditions prevailing in the living body. For this purpose, an artificial saliva solution was used, whose chemical composition is given in Table 1. Temperature of the solution was 37 °C and pH 7. The samples used for the tests had been prepared by cleaning in 96% ethanol using an ultrasonic washer machine for 5 min. The study was started with determination of open circuit potential  $E_{OCP}$ , which is the value at which the process of corrosion begins. Then, the anodic polarization curves were recorded, starting from the value of initial potential. The change of potential occurred in the direction of the anode at a rate of 1 mV/s. The return curve was recorded (polarity reversal) after reaching the anodic current density of 1 mA/cm<sup>2</sup>. On the basis of anodic polarization curves the characteristic parameters describing the resistance to pitting corrosion have been determined: breakdown potential  $E_b$  (mV), repassivation potential  $E_{cp}$  (mV), polarization resistance  $R_p$  (kΩcm<sup>2</sup>), corrosion current density  $i_{corr}$  (μA/cm<sup>2</sup>).

geometry. Ni-Ti alloy tested complied with the requirements of the F 2063-00 standard in terms of chemical and phase composition. The structure of Ni-Ti shape memory alloy, both in the working length part and in the handle, consisted of martensite plates with different orientation of single grains of the parent phase and the presence of twins with single plates of martensite was stated for both new and used files (Fig. 2).

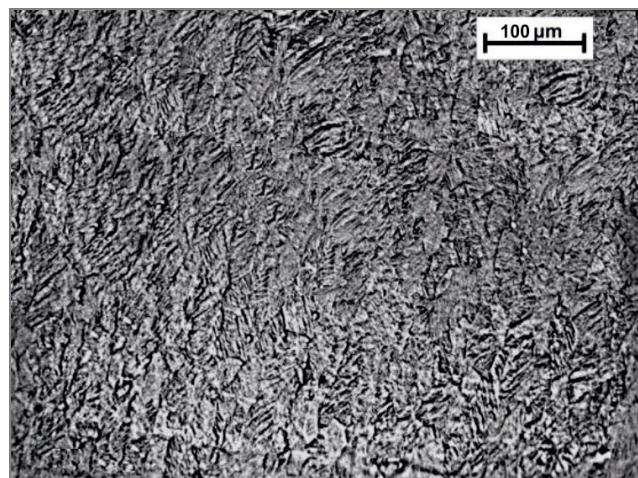


Fig. 2. Martensitic structure of Ni-Ti alloy, new Mtwo 20/06 endodontic file, longitudinal section, 1000× magnification

On the basis of Vickers microhardness measurement the strain hardening the Ni-Ti alloy was eval-

Table 1. The chemical composition of artificial saliva

Chemical composition	Na <sub>2</sub> HPO <sub>4</sub>	NaCl	KSCN	KH <sub>2</sub> PO <sub>4</sub>	NaHCO <sub>3</sub>	KCl
Quantity, g/l	0.260	0.700	0.330	0.200	1.500	1.200

In order to identify possible damage the observation of the surface of the new endodontic files and after six times of use, before and after the corrosion test, was carried out in a scanning electron microscope ZEISS SUPRA 35. The microscope was also equipped with an energy dispersive X-ray spectrometer EDAX. The magnification range was 80×÷1500×, and voltage equal to 20 kV.

### 3. Results

The study of metallographic microscopy revealed the presence of non-metallic inclusions, which were uniformly distributed all over the volume of the files

ated. The tests were carried out for the working part across and along the axis of endodontic files. The results differed from each other in the range from a few to several units of HV. In the transverse section minimum value of 352 HV for the new file 15/05 and maximum value of 370 HV for the used file 20/06 were obtained, Fig. 3. A bit higher values were obtained in the longitudinal section of the files. The minimum value of 360 HV, and the maximum of 388 HV were stated for the new files 15/05, Fig. 4. For the used endodontic files the higher values of microhardness were obtained (both for the white files 15/05 and for the yellow files 20/06). This indicates that alloy strengthening increases while using the endodontic instruments, both in the surface layer and in the core.

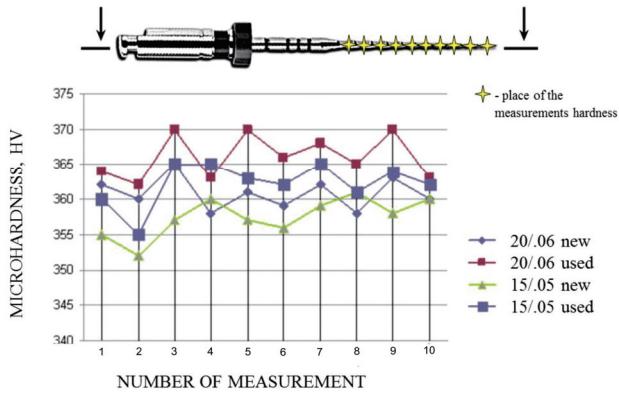


Fig. 3. Change of microhardness on the longitudinal section of the work part of Mtwo files

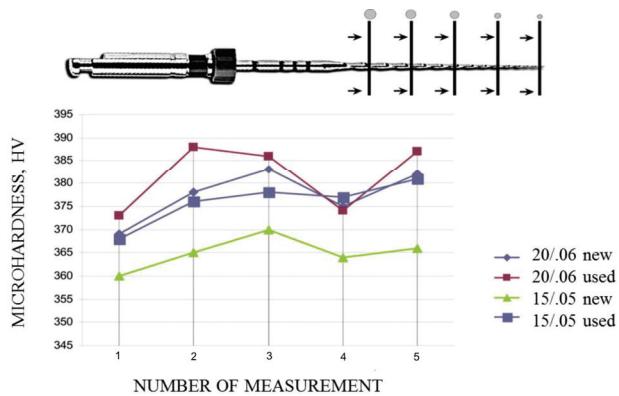


Fig. 4. Change of microhardness on the transverse section of the work part of Mtwo files

In order to evaluate the resistance to pitting corrosion of the endodontic files the test were carried out in environment of artificial saliva. The results showed the difference in resistance to pitting corrosion for the new and the used files, Table 2. Analysis of the results showed that the value of the open circuit potential  $E_{OCP}$ , both for the files 15/05 and 20/06 new and for the used endodontic files, was established after 15 min. The values of potential higher by approximately +90 mV were obtained for the used files compared to the new ones. The corrosion potential  $E_{corr}$  was in the range  $-174 \div +2$  mV for the new files and  $-89 \div -59$  mV for the used ones. The differences observed for the corrosion potential may be caused by the non-uniform degree of wear-out failure of the cutting edges of the files. Higher values of the breakdown potential in the range of  $E_b = +1329 \div +1342$  mV were obtained for the used files, while for the new ones the values of  $E_b = +552 \div +648$  mV were obtained. The repassivation potential reached similar values for the used files ( $E_{cp} = +1113 \div +1124$  mV), while the new files did not show repassivation ability. As a result of repassivation the pitting corrosion process was inhibited, and it follows that the used files exhibit better resistance to pitting corrosion than the new ones. Corrosion current density for the files in size group of 15/05 has smaller values ( $i_{corr} = 0.001 \div 0.003 \mu\text{A}/\text{cm}^2$ ) than for files in size group

Table 2. The results of potentiodynamic research

File	Corrosion potential $E_{corr}$ , mV	Pitting potential $E_b$ , mV	Repassivation potential $E_{cp}$ , mV	Corrosion current density $i_{corr}$ , $\mu\text{A}/\text{cm}^2$	Polarisation resistance $R_p$ , $\text{k}\Omega\text{cm}^2$
15/05 new	+1.7	+647	NO	0.003	7900
15/05 used	-89	+1342	+1113	0.001	17800
20/06 new	-175	+552	NO	0.042	616
20/06 used	-59	+1329	+1124	0.004	6330

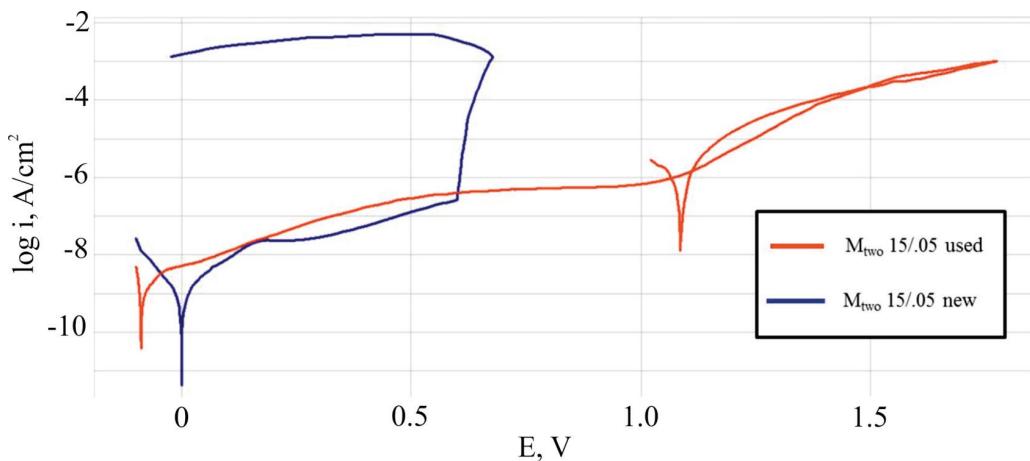


Fig. 5. Example of changing the value of current density as a function of the potential in the logarithmic form for new and used Mtwo 15/05 files

20/06 ( $i_{corr} = 0.004 \div 0.042 \mu\text{A}/\text{cm}^2$ ). Polarization resistance for the files 15/05 was in the range  $R_p = 17800 \div 7900 \text{ k}\Omega\text{cm}^2$ , for files 20/06 the values were smaller and equal to  $R_p = 6330 \div 616 \text{ k}\text{Wcm}^2$ . (Fig. 5).

In order to identify possible damage caused by the usage and corrosion environment impact, the observations of new and used files in scanning electron microscope were also conducted. The observations

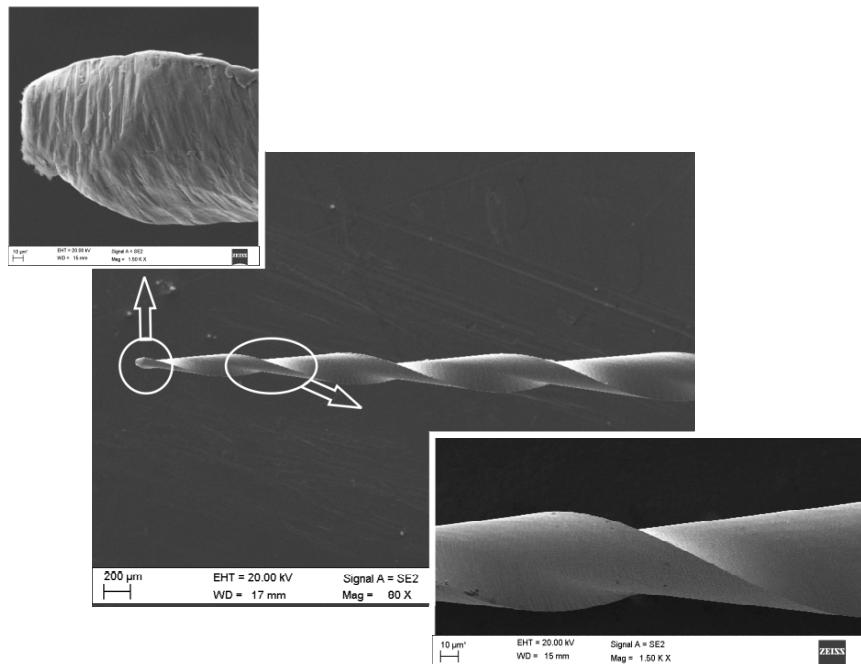


Fig. 6. The surface of the Mtwo 15/05 file in the initial state, 80 $\times$  and 1500 $\times$  magnification

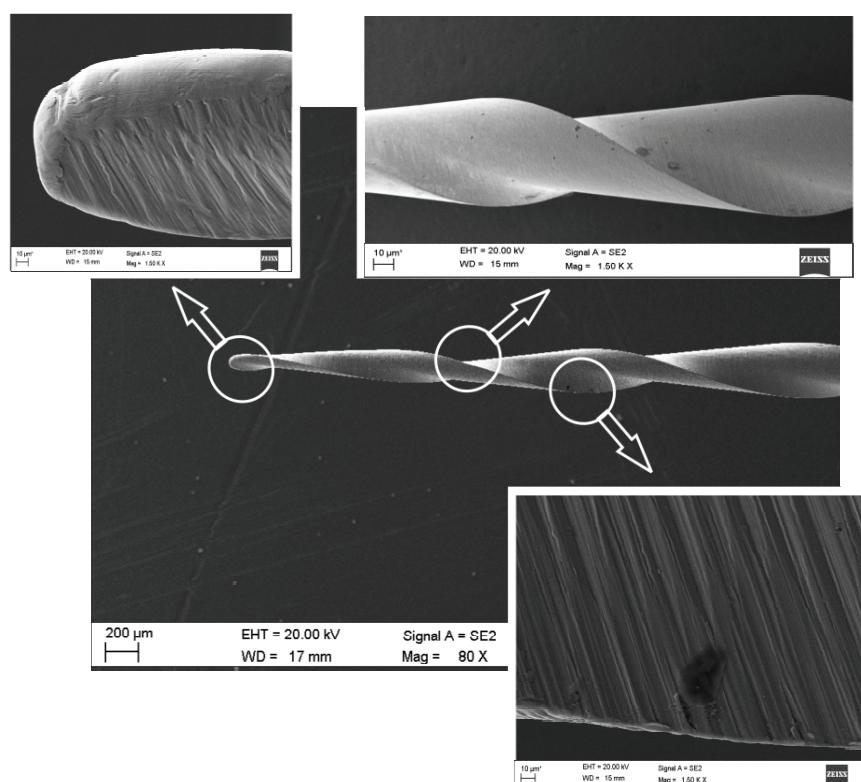


Fig. 7. The surface of the used Mtwo 15/05 file before the corrosion resistance test, 80 $\times$  and 1500 $\times$  magnification

carried out before the corrosion resistance tests showed irregular surface of the files (new and used) in the form of pits and scratches, Fig. 6 and Fig. 7. Visible defects were located along the entire length of the working part of tools. On the surface of the used files blunting the edge of the blade and uneven wear of the

work part over its entire length were observed. On the cutting surface of the new files 15/05 numerous pits were observed, Fig. 8. For the new files 20/06 the chipping of the tip part of the tools was observed after the corrosion tests, Fig. 9. Any pits were reevaluated on the used files, Fig. 10.

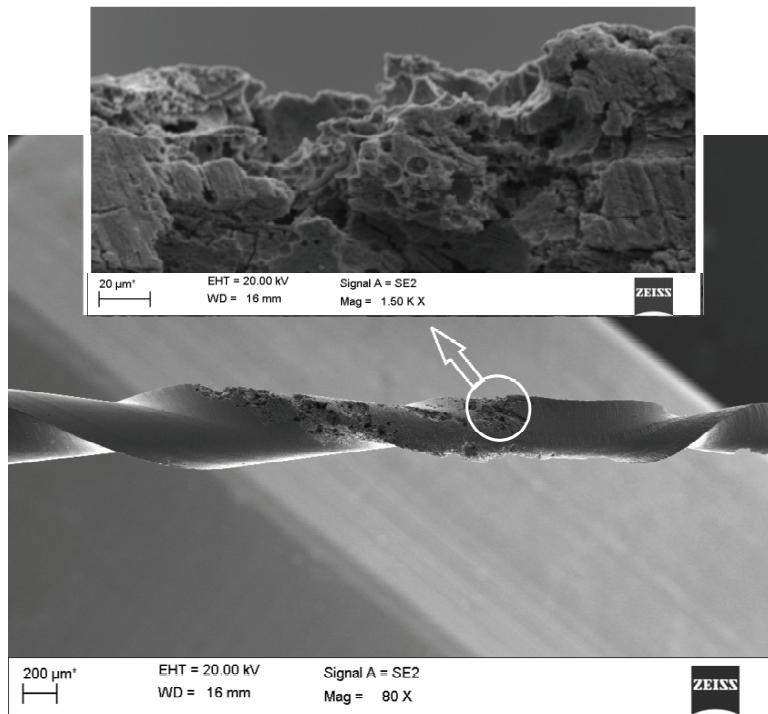


Fig. 8. The surface of the Mtwo 15/05 file in initial state after corrosion resistance test, 80 $\times$  and 1500 $\times$  magnification

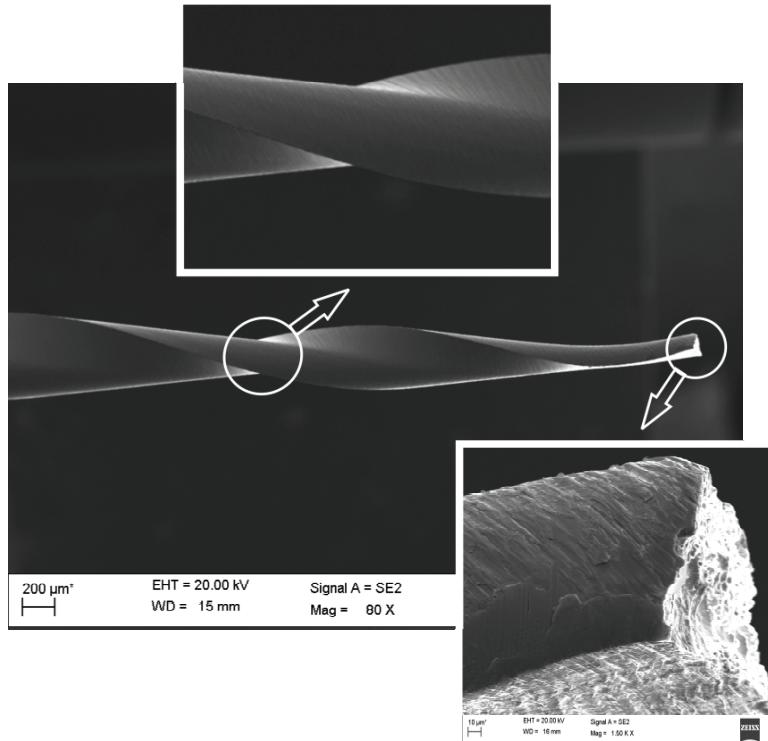


Fig. 9. The surface of the Mtwo 20/06 file in initial state after corrosion resistance test, 80 $\times$  and 1500 $\times$  magnification

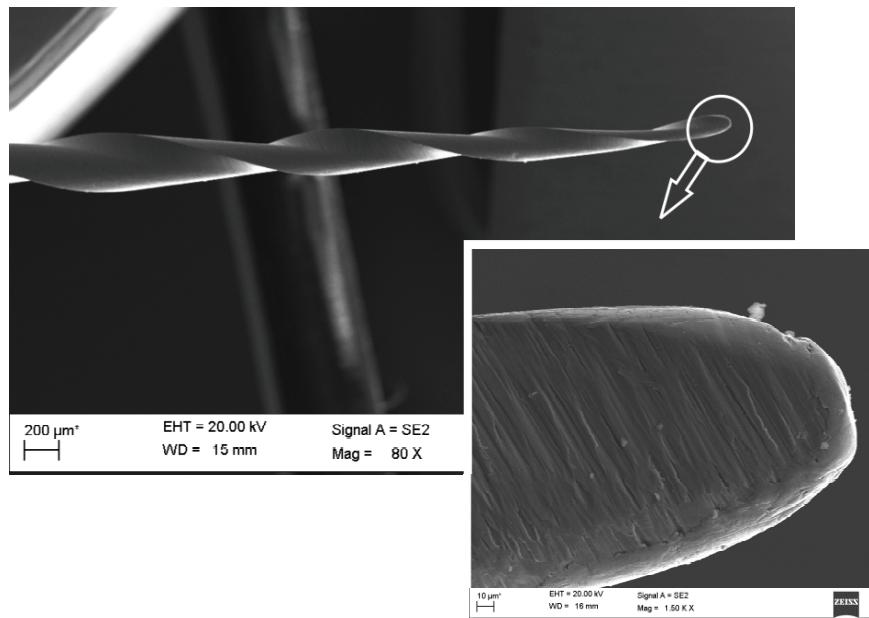


Fig. 10. The surface of the used Mtwo 20/06 file after corrosion resistance test, 80 $\times$  and 1500 $\times$  magnification

## 4. Discussion

The literature data indicate that the superelastic effect is beneficial to the correct treatment process of tightly and curved root canals [4], [7], [12], [14], [19], [23], [25]. Plotino et al. [13] evaluated the effect of autoclave sterilization on cyclic fatigue resistance of rotary endodontic instruments, including the Mtwo files. These studies showed that repeated cycles of autoclave sterilization do not seem to influence the mechanical properties and cyclic fatigue resistance of Mtwo files. However, the tests showed the influence of the usage conditions and sterilization process of Mtwo endodontic instruments made of Ni-Ti alloy on the mechanical properties, microstructure, corrosion resistance and changes in the quality of the cutting surface. The observations of new and used Mtwo files in a scanning electron microscope showed irregular surface of endodontic files in the form of pits and scratches, which is consistent with the results of studies carried out by Eggert et al. [7]. They analyzed the structure of LightSpeed tools in scanning electron microscope and stated that damage of the working blade in the form of pitting is visible both on the used tools and on the new ones. Our studies also showed the visible defects which were located along the entire length of the working part of tools, while on the surface of the used files the blunting of blade edge and uneven abrasion of the working part over its length were observed. On the cutting surface of new endodontic file the corro-

sion pits were observed and chipping of the tip of endodontic instruments occurred. In the used files there were no signs of pitting corrosion. Shahi et al. [22] evaluated the electrochemical corrosion resistance for new files after sterilization, and for Mtwo files used 2 to 8 times by immersing them in 2.5% NaOCl or in saline. They showed that the NaOCl solution does not significantly influence the corrosion resistance of Mtwo files. Numerous research groups have observed the negative influence of multiple sterilization processes on the corrosion resistance of nickel-titanium endodontic instruments. However, Shahi et al. [22] observed that the sterilization cycles of Mtwo files cause an increase of the corrosion resistance. They also noticed a greater corrosion resistance of the used Mtwo files compared to used RaCe files. The electrochemical research carried out in the present work, done in an environment simulating the work of tools during root canal treatment, showed that the wear of cutting edge causes uneven distribution of resisting force which acts on the tool during operation. It has a significant impact on the quality of canal processing and increases the cutting resistance and necessity to use greater forces on files which can result in fracture of the working part of the tool. It was also observed that the used files show better resistance to pitting corrosion than the new ones. In addition, electrochemical studies have shown a greater susceptibility of Mtwo 15/05 and 20/06 files to initiate pitting into the depth of the material for instruments with a larger diameter.

Bhagabati et al. [5] investigated the influence of the manufacturing process of endodontic instruments on the cyclic fatigue resistance. They found that Mtwo files, produced with the traditional grinding process, were significantly less resistant to fatigue than Twisted File (TF) [6]. However, we also evaluated the mechanical properties and concluded that the Mtwo files after being used six times have a greater strain hardening of the zones in the subsurface relative to the core, which is caused by operation process of the tools.

## 5. Conclusions

The research carried out showed the influence of the usage conditions and sterilization process of Mtwo endodontic instruments made of Ni-Ti alloy on the mechanical properties, microstructure, corrosion resistance and changes in the quality of the cutting surface. The results showed that using the tool six times causes damage at the edges of the blades and may cause a lack of continuity of the cutting line resulting in the uneven distribution of the resistive force which acts on the tool during operation. It should be remembered that the research conducted refers only to the two dimensions of the Mtwo files. In order to determine definitive conclusions further studies with the use of other dimensions of the files and evaluation of the resistance to other types of corrosion will be carried out.

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