SUFACE AND XPS STUDIES OF THE NiTi ENDODONTIC ROTARY INSTRUMENTS AFTER MAGNETOELECTROPOLISHING

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Introduction

During manufacturing of NiTi endodontic files the good quality and adequate finishing are of concern. Electropolishing is one method commonly used to modify the metal and/or alloy surface to make it more bio- and haemocompatible [1-5]. Near equiatomic NiTi alloy has become ubiquitous among biomedical materials used for endodontic instruments due to its mechanical (shape memory and superelasticity), corrosion resistance properties, and, most importantly, biocompatibility.
The main aim of the study was to reveal the effects of a new electropolishing process carried out under a constant magnetic field, termed as magnetoelectropolishing (MEP). The primary results of MEP referred to Nitinol samples have been presented by the authors elsewhere [1, 6-9]. In this work we investigated Nitinol rotary endodontic instruments (**Fig. 1**) by surface interferometry and XPS results change after MEP, the process which affects also mechanical properties like the bending and fatigue resistance.

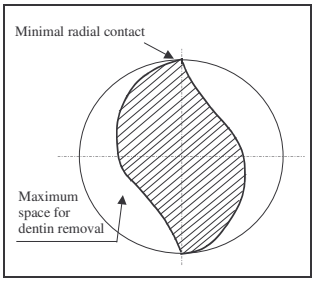

Fig. 1. Typical cross-section of NiTi endodontic rotary instrument

Experimental

Materials and set-up
Two sets of M_{two} NiTi endodontic files [10] were subjected to the study, the first one, covering as-received (AR) and magnetoelectropolished MEP samples, and the second one obtained after EP and MEP. Magnetoelectropolishing of working surface of NiTi endodontic instruments was performed under the electrochemical conditions referred to Nitinol and presented by the authors elsewhere [8, 9].

Apparatus and procedures
The surfaces of NiTi endodontic files were studied by interferometric method with Taylor-Hobson Precision Talysurf CCI 6000 (Coherent Correlation Interferometry) [11]. It is a powerful instrument allowing for getting multiple surface characteristics, together with digital data concerning *Sa* and *Sz* [12].

The XPS (X-ray Photoelectron Spectroscopy) measurements on EP, and MEP of Nitinol files samples were performed using SCIENCE SES 2002 instrument. The studies were carried out under standard conditions within the binding energy of −1100 eV to 0 eV. For the XPS analyses the CasaXPS 2.3.14 programme was used.

Results and Discussion

The surface roughness analysis, concerning both as-received (AR) and magnetoelectropolished (MEP) Nitinol endodontic files, was carried out using the interferometric method. The 3D NiTi endodontic file surface of AR original and MEP are presented in **Fig. 2**.

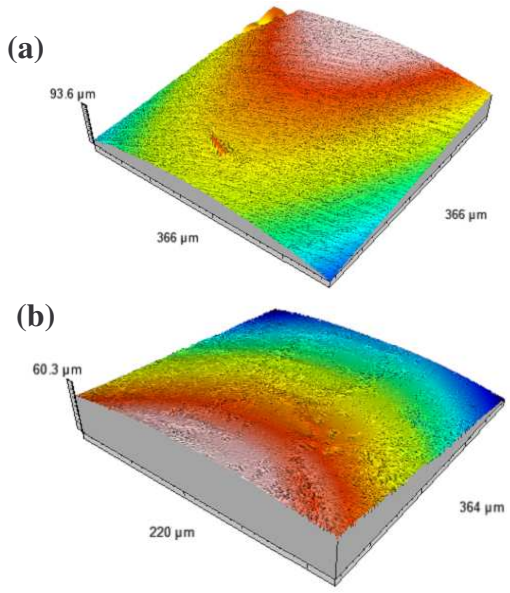

Fig. 2. 3D endodontic file surface interferometry: (a) as-received AR, (b) after magnetoelectropolishing MEP

Digital values of parameters *Sa* and *Sz* of NiTi endodontic files AR and MEP are presented in **Table 1**. A dramatic change in *Sz* from 85.1 of AR to 9.3 of MEP on surface after form removal was observed.

Table 1. Numerical results of interferometry studies

Parameter	Data of endodontic file surface, µm			
	As received **AR**	**After form removal**	**MEP**	**After form removal**
Sa	12.7	0.715	11.5	0.737
Sz	93.6	85.1	60.3	9.3

XPS studies on NiTi endodontic instruments electro-polished EP and MEP have been carried out within the binding energy −1100 eV to 0 eV and the general results are presented in **Fig. 3**.

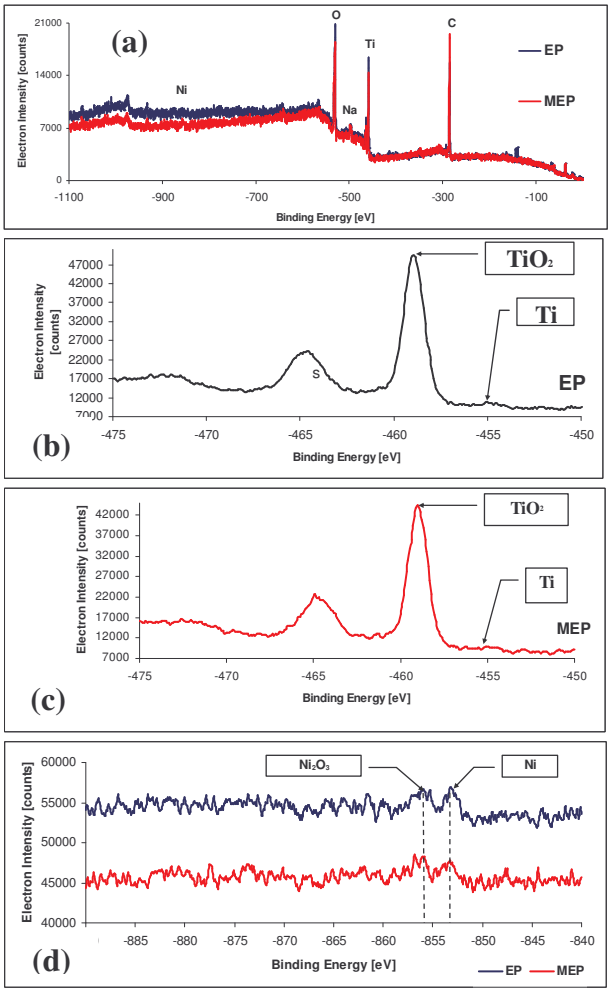

Fig. 3. XPS spectra of Nitinol endodontic file: (a) survey of surfaces after EP and MEP, (b) titanium and Ti oxides after EP, (c) titanium and Ti oxides after MEP, (d) nickel and Ni oxides after EP and MEP comparison

In **Fig. 4** the beneficial effect of MEP is visible. The contents of Ni compounds (see Fig. 3) is higher after EP (18.3%) than after MEP (10.2%), whereas the contents of Ti compounds is higher after MEP (83.4%) than after EP (76.6%). In Fig. 4 the Ti/Ni ratio indicates almost double surpass of titanium over nickel in the surface film after MEP in comparison with the total amount of that ratio after EP.

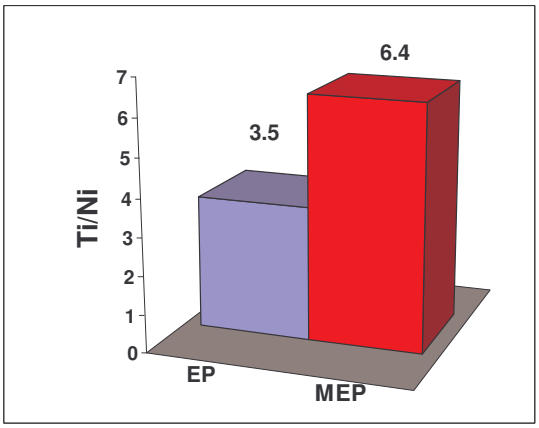

Fig. 4. Ti/Ni ratio comparison of Nitinol endodontic file after EP and MEP finishing

Conclusion

The investigation results obtained indicate a considerable improvement of MEP surface in comparison with AR and EP surface. The results should serve also on the way to reduction of the high incidence of breakage of engine-driven files during clinical use.

Acknowledgements

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