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Corrosion resistance of diamond-like carbon coated rotary endodontic instruments - pilot study

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Summary:

Abstract

Aim: The purpose of this study was to determine if a diamond-like carbon (DLC) coating in NiTi alloy engine-driven endodontic files (Hyflex CM, Hyflex EDM and Reciproc Blue) can protect the surface against sodium hypochlorite (NaOCl) driven corrosion.

Materials and Methods: A total of 12 files were split in six groups: 2 Hyflex CM files (size 25, .06 taper, 25 mm), 2 Hyflex EDM files (size 25/~ OneFile, 25mm), 2 Reciproc Blue files, 2 Hyflex CM (size 25, .06 taper, 25 mm) files with DLC coating, 2 Hyflex EDM files (size 25/~ OneFile, 25mm) with DLC coating and 2 Reciproc Blue files with DLC coating. One file from each group was subject to two NaOCl immersion protocols, with surface analysis performed in between through scanning electron microscopy (SEM), and the other was left untouched as received by the company. In the first experimental NaOCl protocol, files were dynamically immersed for 5 minutes in 4 ml of 6% sodium hypochlorite solution at 37 ºC. Subsequently, files were immediately submitted to dynamic immersion in distilled water for 1 minute and air dried. Following SEM, in a second immersion protocol, the previously immersed files were briefly immersed in 4 ml of 6% sodium hypochlorite solution, immediately removed, and left untouched for 1 hour, after which all files were rinsed with distilled water and air dried. All files were observed in SEM. Additionally, two files (Hyflex EDM with and without DLC coating) were submitted to cyclic fatigue in a specifically developed device.

Results: Uncoated Hyflex CM file showed a uniform texture with machining grooves, preserved after coating, and loss of DLC coating material on the cutting edge of the instrument after the second immersion protocol, while with the Hyflex EDM system the main noticeable change was in the coated EDM file which revealed a less pronounced texture, with the pits, pores and voids in the inner section of the coil having a smoother relief. Surface analysis of uncoated Reciproc Blue revealed a uniform texture with machining grooves, also preserved by DLC coating, and no signs of failure to resist the immersion protocol in both observations. Cyclic fatigue resistance testing showed signs of wear on the cutting edge of the Hyflex EDM uncoated instrument and extensive loss of coating on the inner section of the coil of the DLC coated file with the edge appearing to be better preserved in the latter.

Conclusions: Our findings suggest there’s no difference concerning sodium hypochlorite driven corrosion resistance among files with diamond-like carbon coating or in its original surface.

Key-words: corrosion resistance; cyclic fatigue; DLC coating; engine-driven endodontic files; nickel-titanium; sodium hypochlorite.

Resumo:

Objetivo: Este estudo pretende avaliar se um revestimento de diamond-like carbon em limas endodônticas mecanizadas de Níquel-Titânio (Hyflex CM, Hyflex EDM and Reciproc Blue) tem um efeito protetor da superfície contra corrosão por contacto com hipoclorito de sódio (NaOCl).

Materiais e métodos: Um total de 12 limas foram divididas em 6 grupos: 2 limas Hyflex CM, 2 Hyflex EDM, 2 Reciproc Blue, 2 Hyflex CM com revestimento DLC, 2 Hyflex EDM com revestimento DLC e 2 Reciproc Blue com revestimento DLC. Uma lima de cada grupo foi sujeita a dois protocolos de imersão em NaOCl com análise de superfície através de microscopia eletrónica de varrimento realizada após o primeiro e previamente ao segundo protocolo. A segunda lima do grupo foi mantida tal como fornecida pelo fabricante. No primeiro protocolo de imersão, cada lima foi dinamicamente imersa durante 5 minutos em 4 ml de hipoclorito a 6% e a uma temperatura de 37ºC. De seguida, a lima foi retirada e dinamicamente imersa em água destilada durante 1 minuto, com secagem posterior. Após a análise de superfície, as limas foram sujeitas ao segundo protocolo de imersão, no qual foram imersas em hipoclorito a 6% e retiradas imediatamente a seguir. Posteriormente, foram colocadas num suporte sem qualquer manipulação durante 1 hora, após a qual foram lavadas com água destilada e posterior secagem. Todas as limas foram novamente analisadas através de microscopia eletrónica de varrimento. Adicionalmente, duas limas (Hyflex EDM com e sem revestimento DLC) foram sujeitas a fadiga cíclica num dispositivo especificamente desenvolvido para o efeito.

Resultados: A lima Hyflex CM mostrou uma textura de superfície uniforme com marcas de maquinação visíveis, tendo essa textura sido mantida após o revestimento de superfície com DLC. Após o segundo protocolo de imersão, a lima deste sistema com revestimento mostrou perda de DLC na superfície de corte da mesma. Relativamente ao sistema Hyflex EDM, a distinção mais notável foi uma suavização do relevo na porção interna da hélice das limas após o revestimento. A análise de superfície do sistema Reciproc Blue mostrou uma textura de superfície uniforme com marcas de maquinação, tendo também o relevo da superfície sido mantido após a adição do revestimento de DLC, tendo este também demostrado sinais de resistência aos protocolos de imersão em hipoclorito visto não se ter verificado perdas de material em nenhuma das observações. O teste de resistência à fadiga cíclica revelou sinais de desgaste na aresta de corte da lima do sistema Hyflex EDM sem revestimento e perda significativa de material de revestimento na porção interna da hélice da lima revestida, ainda que a aresta de corte permanecesse mais integra (também esta com perda de material de revestimento).

Conclusões: Os resultados do presente estudo sugerem que não se verifica uma diferença apreciável entre limas revestidas e não revestidas com diamond-like carbon no que concerne à resistência à corrosão decorrente do contacto destes instrumentos com hipoclorito de sódio.

Palavras-chave: fadiga cíclica; hipoclorito de sódio; limas endodônticas mecanizadas; níquel-titânio; resistência à corrosão; revestimento DLC.

Introduction:

Since their introduction, nickel-titanium (NiTi) instruments have become increasingly popular not only among specialists, but also generalists (1). The popularity and acceptance achieved by these materials resulted from a series of specific advantages when applied to endodontic files, such as their increased flexibility, torsional fracture resistance, shape memory effect and stress hysteresis when compared to their predecessors, stainless steel instruments (2). However, preparation errors with engine-driven endodontic instruments may still occur as both materials tend to straighten curved canals, in spite of having been demonstrated that preparation with NiTi endodontic instruments results in less canal transportation and fewer preparation errors (3). The drawback of this alloy resides in the possibility of fracture in use without undergoing any permanent deformation or other visible warning signs (4).

In terms of mechanical behavior, the percentage of each metal and its microstructural phase is preponderant. Conventional NiTi alloy is 56% nickel and 44% titanium (1, 3) and manufacturers influence the properties of the file by changing the microstructural phase of the alloy between its 3 phases (austenitic, martensitic and R-phase) (1). If the alloy is at a temperature higher than the austenitic end temperature, the instrument presents itself with superior superelastic properties, stiff and hard. On the other hand, if the temperature is bellow the martensitic finish temperature, the alloy is soft, ductile, easily deformed and possesses shape memory effect (3).

The rhombohedrally distorted phase (R-phase) appears between martensitic and austenitic phases in a very narrow range of temperatures (1, 3). This phase has particular characteristics that make it useful from a manufacturing perspective, the lower levels of stress needed to induce a plastic deformation, make it particularly suitable for the twisting metal wire manufacturing method, being followed by an austenitic transformation in order to maintain the shape (3). Clinically, these files have proven to be more resistant to cyclic fatigue and more flexible when compared with instruments without this thermal treatment (3).

Conventional NiTi endodontic instruments are composed mainly of an austenitic phase alloy, as the finishing temperature of this phase is below body temperature. They have to be grinded rather than twisted and this may lead to defects on the surface of the file, which have been reported to have a negative influence in the fracture resistance, cutting efficiency and resistance to corrosion (3, 5, 6).

Sodium hypochlorite (NaOCl) is the most commonly used disinfectant in endodontics (7), but it is also known for its corrosive effect and thus, the potential to create extensive damage to NiTi alloy instruments (Fig. 1). Either in clinical use or in the cleaning process, endodontic files are often exposed to the corrosive effect of sodium hypochlorite (7-9). In NiTi alloys, exposure to corrosive solutions leads to “pit nucleation” or “pitting” (7, 8), which consists of the nickel ions removal on the surface of the instrument, leaving a surface irregularity (8) (corrosion pits) prone to propagating into full cracks and eventual separation of the instrument (4).

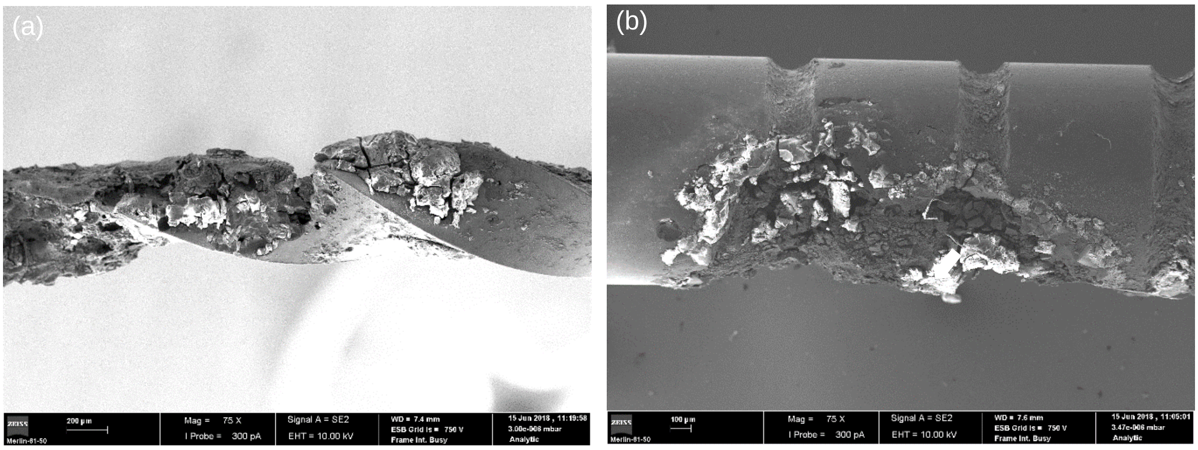

Figure 1: Unmodified Hyflex CM (a) and Hyflex EDM (b) at 75X magnification after exposure to sodium hypochlorite (6% at 60ºC for 12h).

NiTi file manufacturers have been developing and testing additional treatments to improve instrument characteristics, among which it is possible to highlight the thermal, mechanical and surface treatment. Among those, electropolishing emerged as a viable option to smoothen the surfaces by electrochemical removal of material. In this process the surface of the file is subject to an electric current in an electrolyte(s) bath that partially dissolves the surface, especially irregularities (peaks or sharp edges where the current density becomes higher), ultimately altering the surface of the file in composition and texture, leaving the oxide layer more homogeneous, with less relief and residual surface stress, thus improving the resistance of the surface to corrosion (4). The resulting surface shows significant improvement in corrosion (7, 8) and cyclic fatigue resistance (10).

Contrarily to the electropolishing process, chemical vapor deposition (CVD) of diamond-like carbon consists of a chemical reaction that results in an added layer on the surface of the coated material. That layer is composed by artificially synthetized diamond (11) and has been studied for several applications in the medical field for its’ reported advantages, such as lubricity, stability and antibacterial properties (12, 13).

Palma et. al.(14) have previously reported findings of hypochlorite driven corrosion (pitting) in Hyflex CM and Hyflex EDM systems, the purpose of this study is to determine if a DLC coating in NiTi alloy engine-driven endodontic files (Hyflex CM, Hyflex EDM and Reciproc Blue) can protect the surface against that phenomenon.

Materials and Methods:

Review:

A research was made using PubMed Database searching for English papers with the following keywords and Boolean connectors: "(nickel titanium OR niti) AND endodontic AND (fatigue resistance OR corrosion OR heat treatment OR surface treatment)".

Methods:

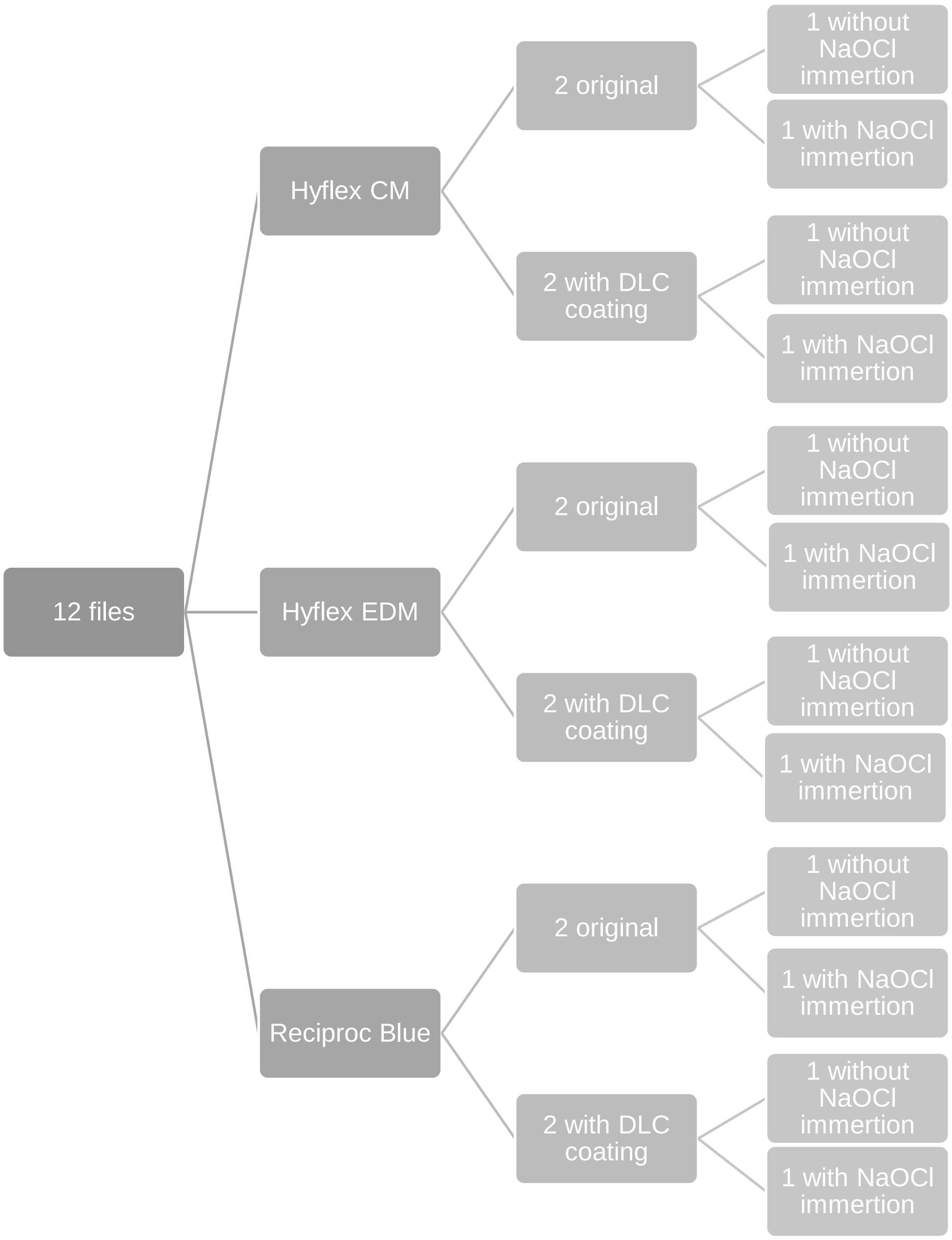

Figure 2: Organogram of the constitution of the experimental groups.

A total of 12 files were split in six groups: 2 Hyflex CM files (size 25, .06 taper, 25 mm) (Coltène-Whaledent, Altstätten, Switzerland) [Group CM1], 2 Hyflex EDM files (size 25/~

OneFile, 25mm) (Coltène-Whaledent, Altstätten, Switzerland) [Group EDM1], 2 Reciproc Blue files (VDW, Munich, Germany) [Group RB1], 2 Hyflex CM files (size 25, .06 taper, 25 mm) (Coltène-Whaledent, Altstätten, Switzerland) with DLC coating [Group CM2], 2 Hyflex EDM files (size 25/~ OneFile, 25mm) (Coltène-Whaledent, Altstätten, Switzerland) with DLC coating [Group EDM2] and 2 Reciproc Blue files (VDW, Munich, Germany) with DLC coating [Group RB2]. DLC coating was made by CVDentus©. (Fig. 2)

In each group one file was subject to two experimental NaOCl immersion protocols, with surface analysis in between, and the other was left untouched as received by the company (Fig. 3).

All files were analyzed through scanning electron microscopy (SEM) with a field emission gun (FEG) microscope (Gemini 2, Zeiss, Oberkochen, Germany).

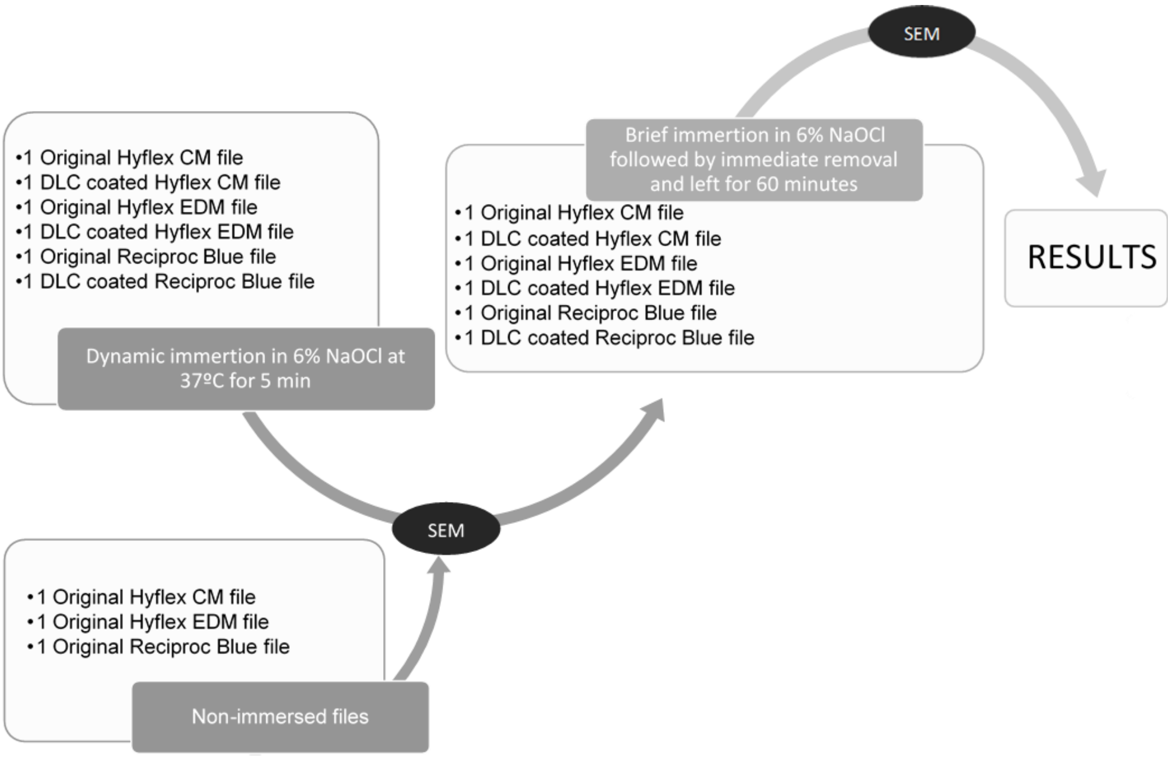

Figure 3: Workflow of the experimental protocol.

In the first experimental protocol, the file was dynamically immersed for 5 minutes in a small container with 4 ml of 6% sodium hypochlorite solution (CanalPro NaOCl, Coltène-Whaledent, Altstätten, Switzerland) at 37 ºC in contact with the whole file (including the shaft). During dynamic immersion, the endodontic instruments were used with an endodontic handpiece (Endodontic Motor CanalPro CL, Coltène-Whaledent, Altstätten, Switzerland), according to the manufactures recommendations (500 rpm for Hyfled CM and EDM systems and 300 rpm for Reciproc Blue). Following the immersion protocol, files were immediately dynamically immersed in distilled water for 1 minute to neutralize the effect of sodium hypochlorite solution, air dried and stored in a labeled plastic container.

From the immersed file of each group and from one of the non-immersed files, two images were taken: the first in the shaft (between 16 and 17mm) with 150x zoom; the second in the cutting flute of the file with 150x zoom.

In a second experimental step, the previously immersed files, were briefly immersed in 4 ml of 6% sodium hypochlorite solution, immediately removed, and left untouched for 1 hour, simulating the time between clinical use and the time spent until washing and sterilization procedures begin. After 60 minutes, all files were rinsed with distilled water, air dried and stored in a labeled plastic container. All files were analyzed again through scanning electron microscopy.

In a parallel pilot trial, two files (one Hyflex CM with DLC coating and one without) were selected to be subject to cyclic fatigue in order to observe the influence of the coating in cyclic fatigue resistance of the instrument, as well as the resistance of the coating itself.

In a device specifically developed for the experiment, aiming to simulate a real clinical situation, two instruments (one Hyflex EDM with DLC coating and one without) were selected, positioned in the handpiece (CanalPro CL, Coltène-Whaledent, Altstätten, Switzerland), inserted into the artificial stainless-steel canal and operated according to the speed recommended by the manufacturer (500 rpm for Hyfled CM and EDM systems and 300 rpm for Reciproc Blue).

This device is composed by a base and a vertical axis with fixation and pecking motion abilities through the handpiece, being able, therefore, to simulate a clinical situation. The artificial canal, incorporated at the base of the test device, manufactured in stainless steel with 16 mm in length and 1,5 mm in diameter, allowed the simulation of a radicular curvature with a 5 mm radius and a 45 º angle.

Both files were left in the device for approximately 9 minutes and then observed through scanning electron microscopy.

Results

Review:

The titles and abstracts of the 138 initially found publications were analyzed and 44 were selected for full text analyses. Seven were ultimately included. Ten cross references were added. Therefore, 17 papers were included in this review.

Methods:

SEM analysis of Hyflex CM files

Superficial analysis conducted by SEM on unmodified CM files showed a uniform texture with machining grooves perpendicular to the long axis of the instrument (Fig. 4a). After coating with diamond-like carbon (DLC) material machining grooves are still visible, thus preserving the surface characteristics (Fig. 4b). Both files presented a significant amount of surface debris (Fig. 4a, b).

After the first immersion protocol (6% sodium hypochlorite solution at 37 ºC during 5 min) files presented less surface debris (fig. 5). No other noticeable differences were observable.

Following the second immersion protocol (brief immersion in 6% sodium hypochlorite solution, immediately removed, and left untouched for 1 hour), no differences were observed in the uncoated file (Fig. 6a) when compared with the previous observation (Fig. 5a). The coated file however, showed loss of coating material on the cutting edge of the instrument (white arrow in Fig. 6b and detail in Fig. 6c, d).

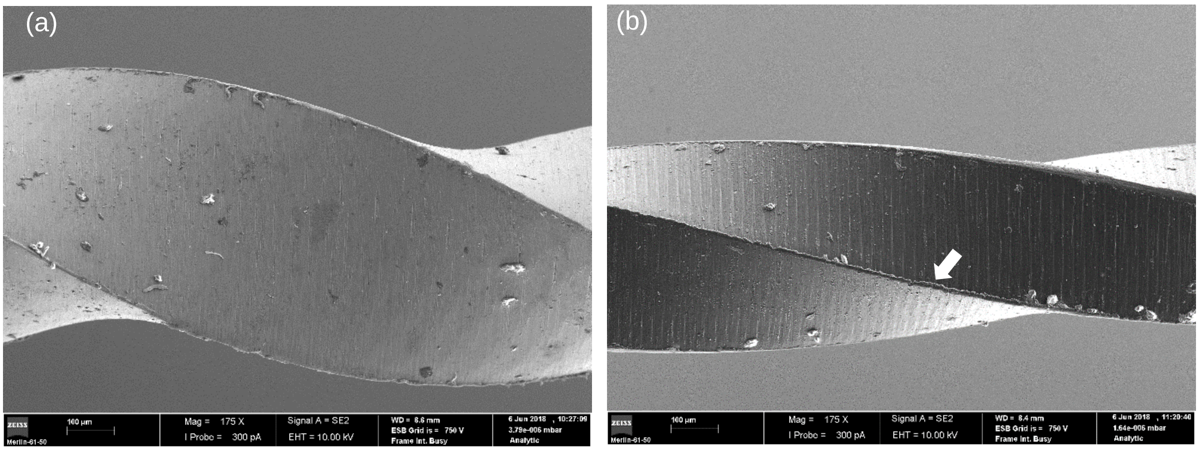

Figure 4: Original Hyflex CM at 175X magnification (a) and with experimental DLC coating (b). White arrow pointing at original coating of the edge of the file.

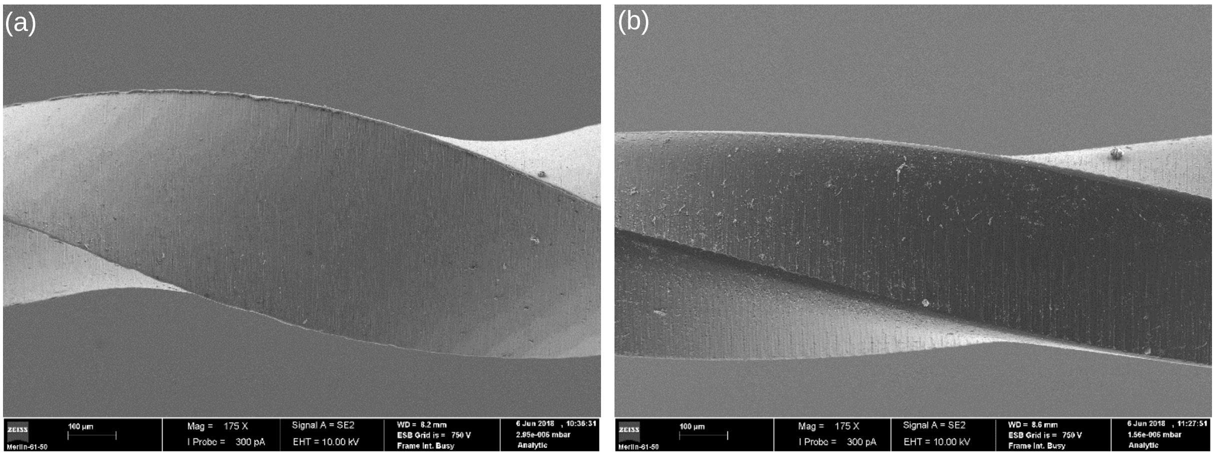

Figure 5: Original Hyflex CM at 175X magnification (a) and with experimental DLC coating (b) after immersion in 6% sodium hypochlorite solution at 37 ºC during 5 min. White arrow pointing at a seeming build up of coating material on the cutting edge.

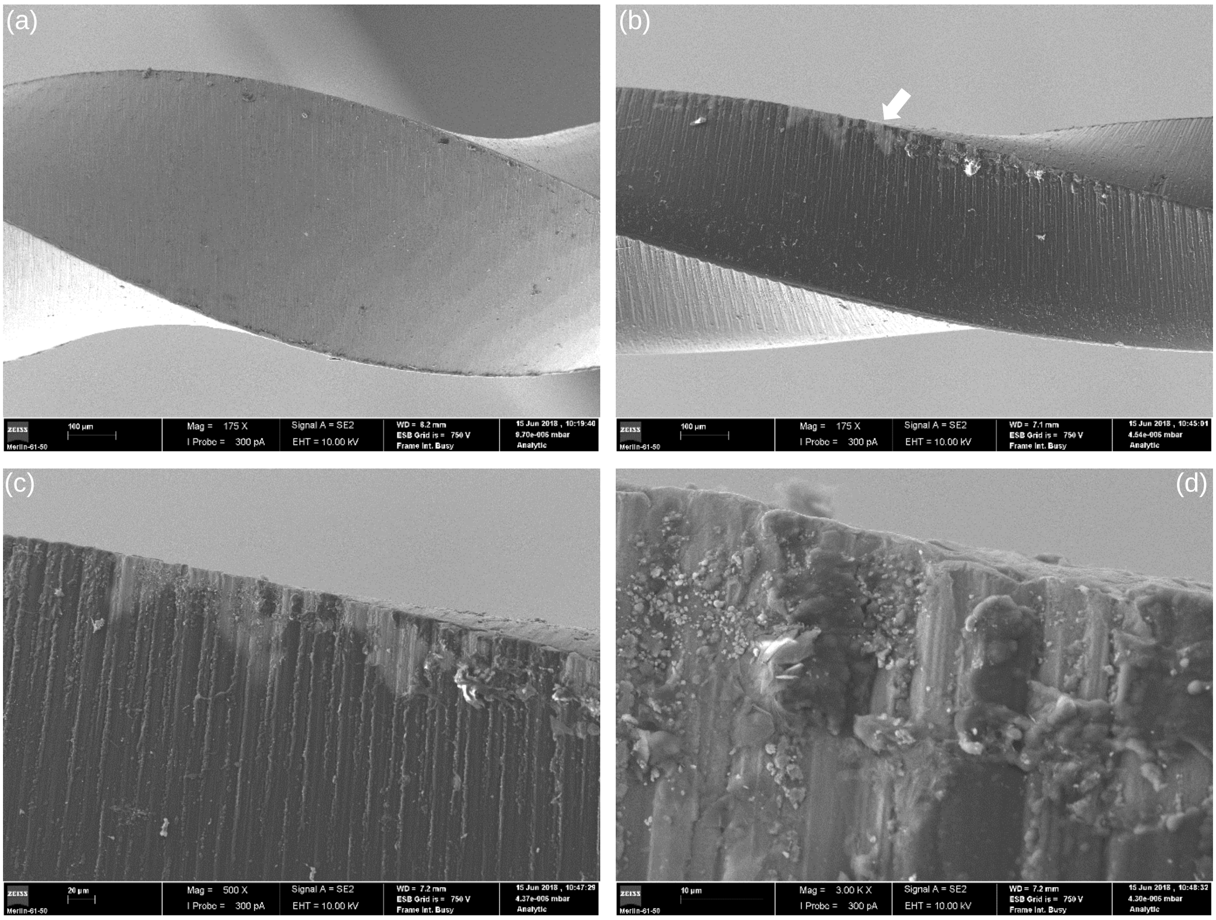

Figure 6: Original Hyflex CM at 175X magnification (a) and with experimental DLC coating (b) after the second immersion protocol (brief immersion in 6% sodium hypochlorite solution, immediately removed, and left untouched for 1 hour) and detail of loss of coating material at higher magnification of 500X (c) and 3000X (d). White arrow pointing at loss of coating material.

SEM analysis of Hyflex EDM files

SEM observation of the Hyflex EDM original files surface shows irregularities characterized by pits, pores and voids (Fig. 7a). The DLC coated instrument shows the same general surface characteristics of the uncoated file but less pronounced, especially on the inner section of the coil (white arrow in Fig. 7b) where the surface appears almost polished without the defining pits, pores and voids.

No noticeable differences were found between the instruments before and after the first immersion protocol, as the uncoated files kept their surface characteristics unchanged as did the coated ones (Fig. 8a, b).

After the second experimental stage, the second SEM analysis revealed, again, no significant differences on the active section of the instrument in both coated and uncoated groups (Fig. 9a, b). However, in the shaft of the coated file, it was possible to observe sighs of loss of DLC material coating (Fig. 9c, d).

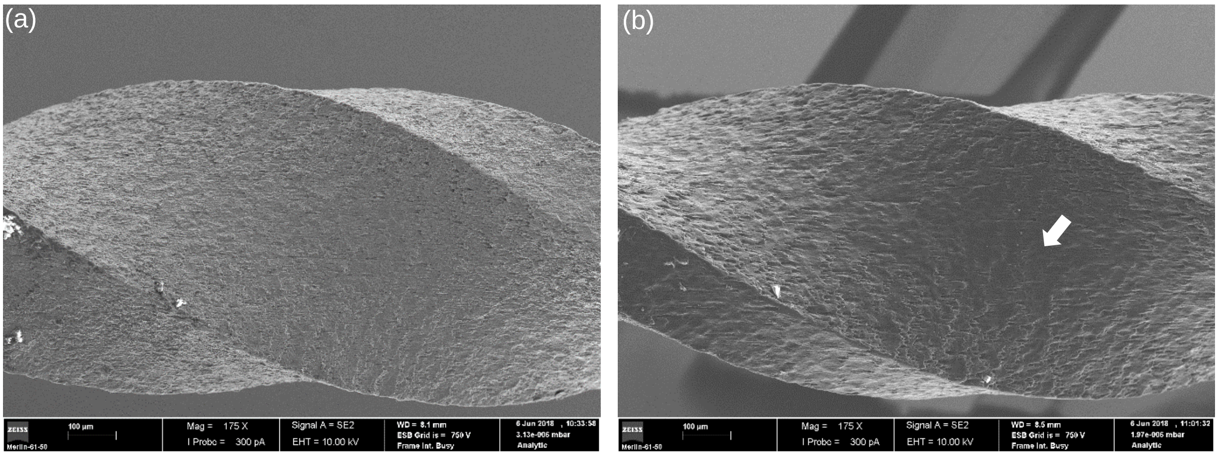

Figure 7: Original Hyflex EDM at 175X magnification (a) and with experimental DLC coating (b). White arrow pointing at less pronounced surface on the inner section of the coil.

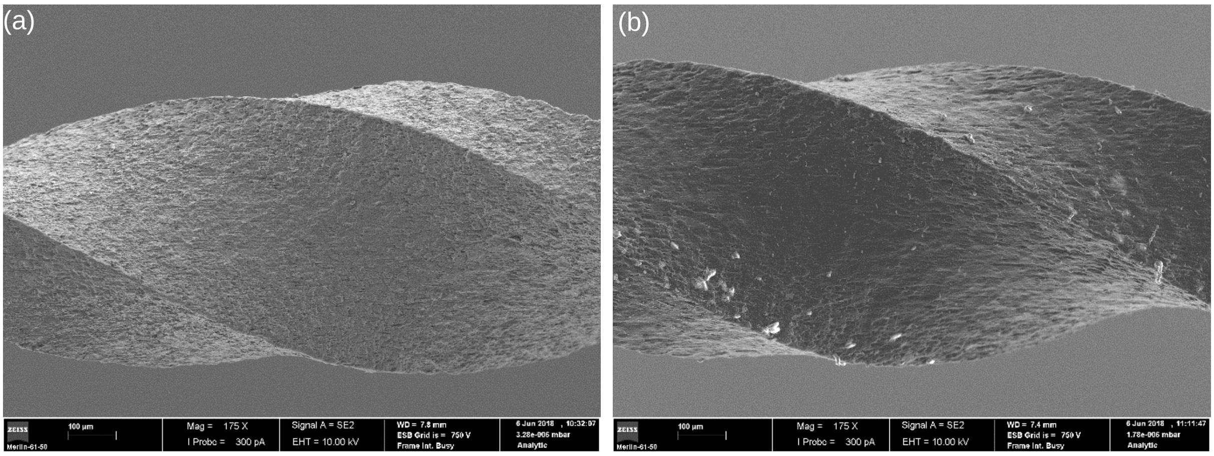

Figure 8: Original Hyflex EDM at 175X magnification (a) and with experimental DLC coating (b) after immersion in 6% sodium hypochlorite solution at 37 ºC during 5 min.

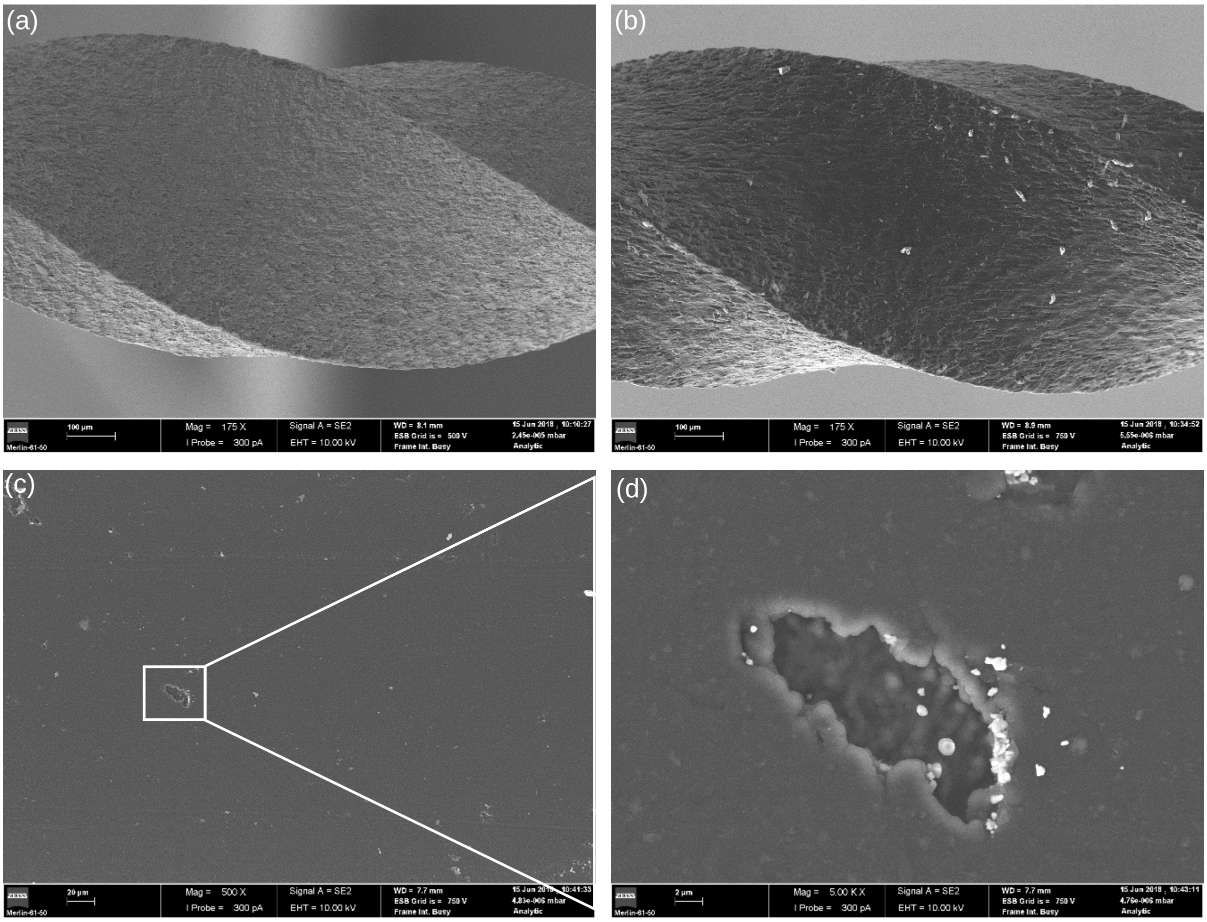

Figure 9: Original Hyflex EDM at 175X magnification (a) and with experimental DLC coating (b) after the second immersion protocol (brief immersion in 6% sodium hypochlorite solution, immediately removed, and left untouched for 1 hour). Detail of shaft localized loss of coating with 500X (c) and 5000X magnification (d).

SEM analysis of Reciproc Blue files

Surface analysis of uncoated Reciproc Blue instruments reveals a uniform texture with machining grooves perpendicular to the long axis of the instrument (Fig. 10a). After coating with diamond-like carbon material, machining grooves are still visible, thus preserving the surface characteristics (Fig. 10b). No clusters of coating material are visible (Fig. 10b).

After the first experimental immersion protocol (Fig. 11a, b) no noticeable differences were observable when comparing with pre-immersion observation.

The final observation (Fig. 12) after the second experimental stage revealed, again, no significant differences between coated and uncoated instruments, as well as between the files subject to the first immersion protocol, with the DLC coating showing no signs of failure to resist the immersion protocol in both observations.

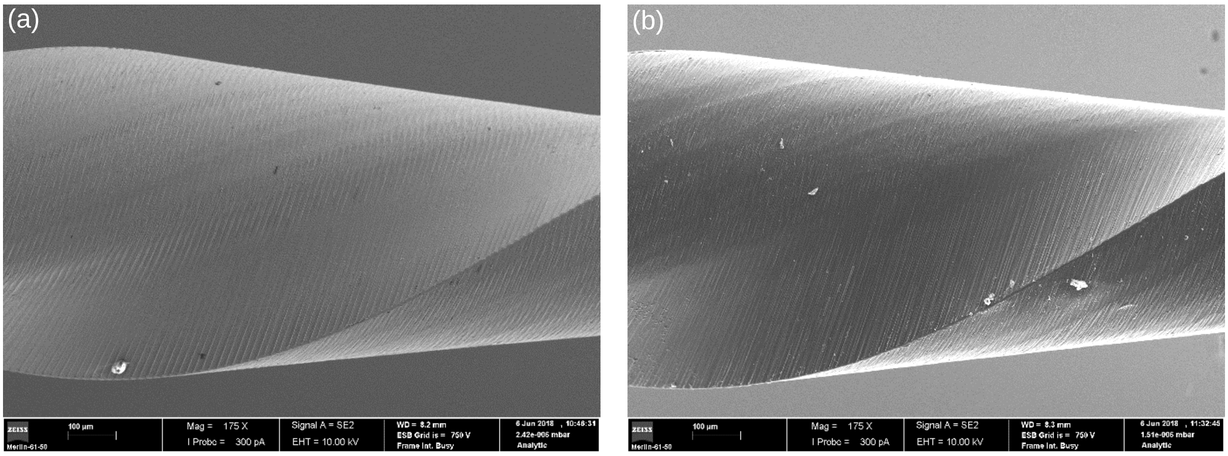

Figure 10: Original Reciproc Blue at 175X magnification (a) and with experimental DLC coating (b).

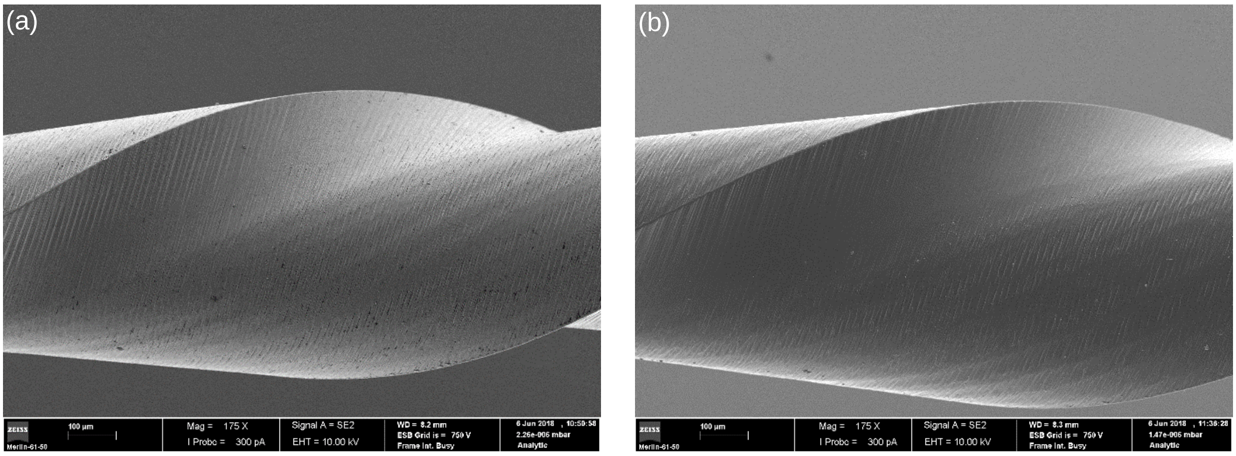

Figure 11: Original Reciproc Blue at 175X magnification (a) and with experimental DLC coating (b) after immersion in 6% sodium hypochlorite solution at 37 ºC during 5 min.

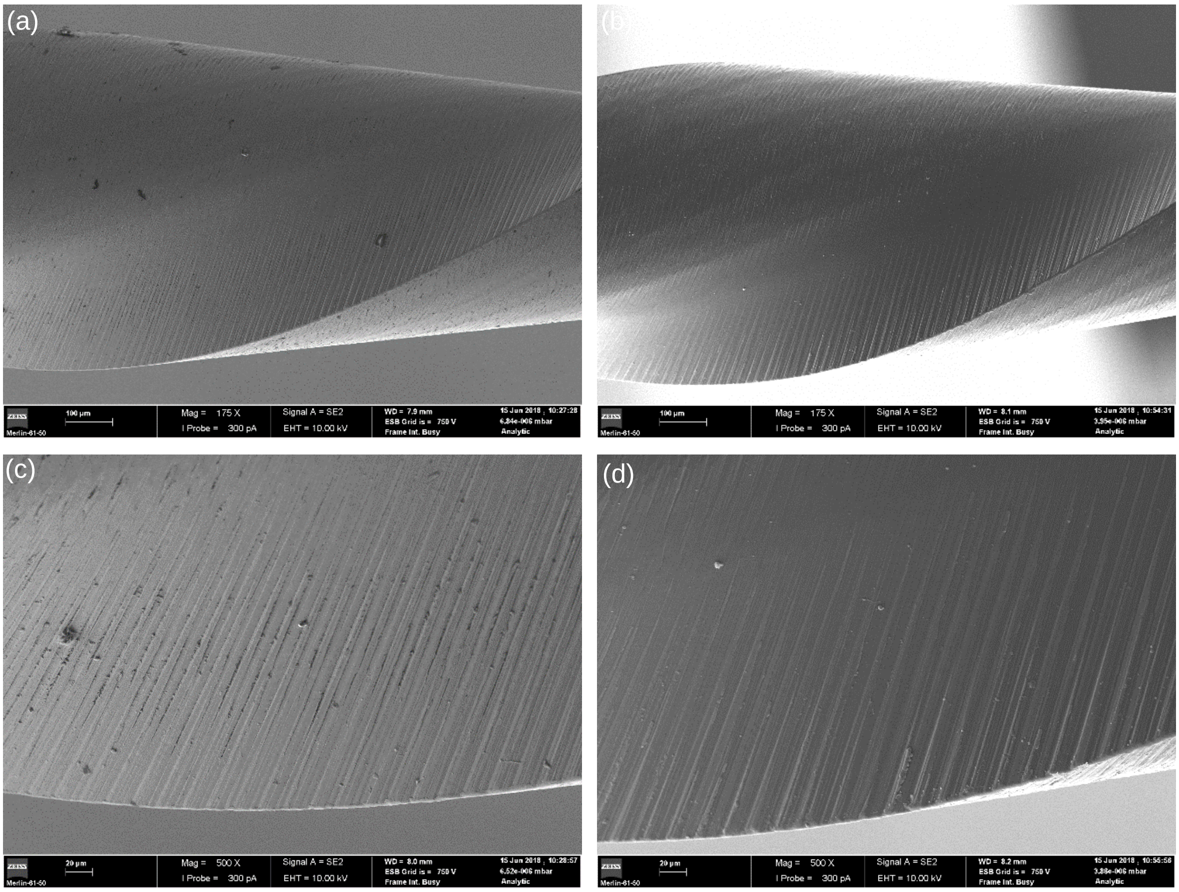

Figure 12: Original Reciproc Blue at 175X magnification (a) and with experimental DLC coating (b) after the second immersion protocol (brief immersion in 6% sodium hypochlorite solution, immediately removed, and left untouched for 1 hour). Detail of cutting edge integrity at 500X magnification on uncoated file (c) and resistance of coating material on the edge of the surface treated file (d).

Cyclic fatigue resistance test files SEM analysis

The unmodified Hyflex EDM subject to the cyclic fatigue protocol shows signs of wear on the cutting edge by presenting a duller edge (Fig. 13a, black arrow). No other noticeable differences from the control (the uncoated and non-subject to the immersion protocol Hylfex EDM from (Fig. 7a)) file were observable.

The coated file presents an extensive loss of coating, more evident on the inner section of the instrument (lighter areas on Fig. 13b). The edge of the cutting section of these instruments seems to have been better preserved, even though it lost a significant amount of DLC coating.

At higher magnifications, numerous microcracks are visible in the original surface of the file after the DLC coating was lost (white arrows at Fig. 13d).

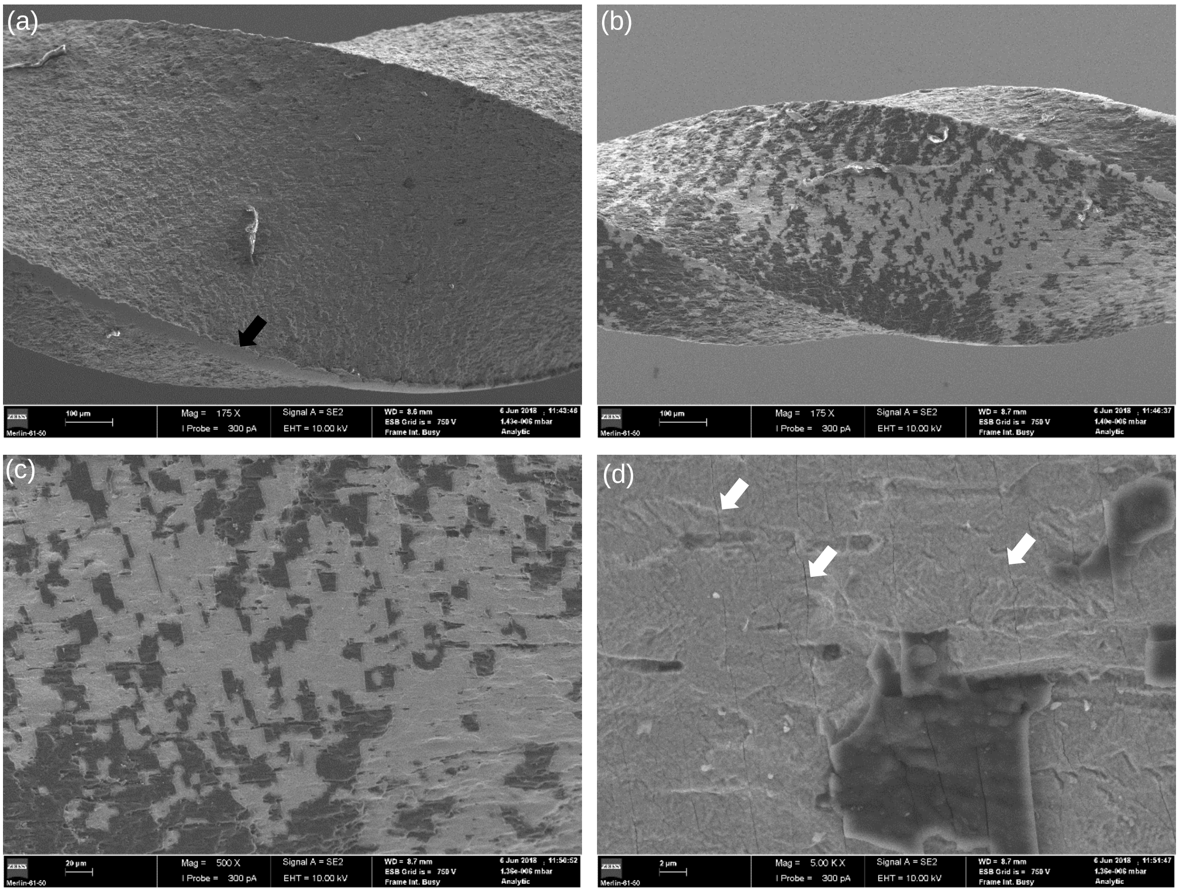

Figure 13: Original Hyflex EDM at 175X magnification (a) and with experimental DLC coating (b) after the cyclic fatigue test protocol. Detail of surface of Hyflex EDM with CVD coating at 500X (c) and 5000X (d). White arrows pinpoint microcracks in the surface of the file after losing the DLC coating. Back arrow points at cutting edge wear.

Discussion

The present pilot study intended to determine if a DLC coating in NiTi alloy engine-driven endodontic files (Hyflex CM, Hyflex EDM and Reciproc Blue) could protect the surface against hypochlorite driven corrosion by studying the morphology and microstructure characteristics of the instrument surface with and without contacting the corrosive solution.

SEM analysis was selected for the evaluation of the file surface because it is a well-documented method to accurately evaluate morphological characteristics.(15, 16)

Hyflex CM SEM results show that the manufacturing process for these files leaves machining grooves in the surface of the instrument, in line with previous reports from Pirani *et. al.*(16). DLC coating conserves the surface texture, indicating that this surface treatment creates an even coating of all the surface irregularities, maintaining its original look. The first immersion protocol resulted in a cleaner surface, free of most debris visible in the non-immersed files. After the second immersion experimental protocol, there's a visible loss of coating material in the edge of the DLC treated file, leaving the underlying metal substrate visible in some points. The effect of the immersion solution on the DLC coating is clear through the progression between the two experimental stages.

The manufacturing process of Hyflex EDM causes a localized melting and partial evaporation of small areas of surface metal that are removed, leaving the observed crater-like finish as previously described by Pirani *et. al.* (16). The DLC treatment left areas with attenuated surface texture, indicating a greater accumulation of coating material in those areas (Fig. 7b). Since these craters transport dentin debris, a smother inner surface of the file might consist an advantage by allowing an easier, more frictionless work. In this mechanized system, the coating was more resistant to removal during the two stages immersion protocol than in the previous one, although there were visible areas with loss of small sections of coating material in the shaft (Fig. 9c, d).

Surface analysis of Reciproc Blue instruments showed a uniform texture, also with machining grooves resultant from the manufacturing process. After coating, the DLC surface treatment preserves the original texture, indicating an even layer of coating material. Through the immersion protocols, both the uncoated and coated instruments seem to resist the immersion solution, with no visible damages in the surface of either the file or the coating.

The analysis of the cyclic fatigue trial instruments show a noticeable wear in the cutting edge of the unmodified file, but this observation needs to take into account that this file was tested in an artificial stainless steel canal, by using metal to test the file (material that it wasn't designed to work on) it is natural to observe unusual amounts of wear.

In the coated file on the other hand, the DLC surface suffered extensive damage and ended up being lost in significant amounts, mainly in the inner section of the file, event that may, once again, be originated by the substrate in which the instruments were tested. The cutting edge is optimized to cut dentin, by using it in a stainless-steel surface, we may be using a sub-optimal cutting angle, thus resulting in the steel fragments being separated when they hit the inner section instead of being immediately split at the edge. This process might be the reason why the inner section of the instrument suffered more wear (shown by the loss of coating) than the edge.

On the other hand, if signs of wear caused by cutting steel are observable and the instrument shows better preservation on the edge of the instrument, the DLC coating may be increasing the cutting ability of the file and the capability of keeping the edge sharp.

The numerous microcracks found in the surface of the Hyflex EDM subject to cyclic fatigue (white arrows at Fig. 13d) are a sign of high cyclic fatigue resistance because the stress is split between all the cracks, therefore diminishing the strain in a localized area (16).

Previous surface treatment solutions, like electropolishing, rely on the removal of surface material to provide an even, more polished surface in order to increase the resistance of the instrument to the corrosive effect of the sodium hypochlorite solution, as described by *Bonaccorso et. al.* (7, 8). The purpose of DLC was to create a non oxidable layer on the surface of the instrument in orther to achieve protection against corrosion and take advantage of the characteristics inherent to the coating material already proven in other aplications, like the antibacterial properties with reports of killing as much as 32.5% of the total bacterial content as described by *Marciano et. al.* (13).

This study, as a pilot study, is limited by its' reduced sample, which made it difficult to observe the pitting effect since this phenomenon has reports of observation as low as 13%, as observed by *Cheung et. al.* (17), low enough so that it did not appear on our sample, but high enough to have clinical relevance, since the same study directly correlates pitting with crack initiation, when present (17).

Conclusions

This study shows no difference in hypochlorite driven corrosion resistance between the tested rotatory systems with diamond-like carbon coating or in its original surface due to the high corrosion resistance observed in the latter. However, the reduced sample size may have a significant effect in the results as the prevalence of pitting reported in the literature may require a higher sample to exhibit the phenomenon. In the cyclic fatigue trial there are some promising results that may indicate a protective effect from the DLC coating.

Further studies in this line of research are needed with higher sample size in other to better determine the effect of DLC coating in hypochlorite driven corrosion resistance of NiTi alloy rotatory systems.

Agradecimentos:

Ao Prof. Doutor Paulo Palma, orientador deste projeto, pela sua disponibilidade e apoio constantes, bem como pelo entusiasmo e dedicação ao ensino. Obrigado pela orientação sempre que me encontrei perdido, obrigado pelo apoio e conhecimentos que de forma tão altruísta se disponibiliza a transmitir, obrigado por embarcar neste projeto e obrigado por me ver merecedor da sua orientação.

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