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CYCLIC FATIGUE RESISTANCE OF NEWLY INTRODUCED SURFACE AND THERMAL TREATED NICKEL-TITANIUM ROTARY FILES

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ABSTRACT

Objectives The main objective of the present study invitro was to evaluate the cyclic fatigue resistance and metallurgic properties of newly thermal treated (CM wire) rotary Ni-Ti files with different surface treatments.

Materials and Methods: A total of one hundred and twenty rotary files were used from three different rotary systems with the same thermomechanical treatment – controlled memory wire (CM Wire) and different surface treatment technique using rotation motion. Groups were set as Group 1: M3 Pro Gold (United Dental, Shanghai, China) (CM Wire) size (25/0.04), Group 2: AF F One Blue (Fanta Dental Material Co., Shanghai, China) [controlled memory wire (CM Wire)] size (25/0.04) and Group 3: Hyflex EDM (25/ 0.04). These files were tested for cyclic fatigue resistance using a custom-made static cyclic fatigue testing apparatus with various angles and radius (angle 60 radius 2.5mm and 5mm radius and angle 90 radius 2.5mm and 5mm). Each rotary file was coated with EDTA after its attachment in handpiece supported in stainless steel cylinder apparatus. M3 Pro Gold rotary files, AF F one blue and Hyflex EDM rotary files were rotated at 500rpm 2.5N/cm torque according to manufacture instructions. Digital watch was used to record the time taken until file fracture in seconds and the (NCF) number of cycles till fracture were calculated. Scanning electron microscope SEM image of the fractured surface were taken. Statistical analysis was performed using one-way analysis of variance and Tukey's test at the 95% confidence level ($P = 0.05$).

Results: After comparing the cyclic fatigue resistance of AF F One Blue and M3 Pro Gold by recording time till the file fracture was ranged between 670s to 1180s in all groups at angle 60 degree with different radius and 10s to 815s in all groups at angle 90 degree with different radius. There were no significant differences between the M3 Pro Gold and AF F One Blue rotary files at angle 60 degree and 90 degree in both radius 2.5mm and 5mm *($P> 0.05$)*, while there were significant differences between the Hyflex EDM and M3 Pro Gold and AF F One Blue rotary files at angle 60 degree, radius 2.5mm *($P> 0.05$)*. Hyflex EDM have the highly significant differences M3 Pro Gold and AF F One Blue rotary files at angle 90 degree, radius 2.5mm ($P < 0.001$).

Conclusions: All new thermomechanical treatment technique - controlled memory CM-Wire rotary systems represented great performance and remarkable enhancement in cyclic fatigue resistance but the Hyflex EDM rotary files had the highest cyclic fatigue resistance among the M3 Pro Gold and AF F one Blue rotary files. Also surface treatment of the rotary files enhancing the cyclic fatigue resistance by decreases surface micro cracks and the tendency to fracture.

Keywords: *Metallurgic property, Controlled memory wire, Hyflex EDM, M3 Pro Gold, AF F One Blue, thermomechanical treatment*

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INTRODUCTION

The Nickel-titanium (NiTi) rotary files are widely used now a days for shaping of root canal with different canal curvature. The unique properties of this alloy improves flexibility of NiTi rotary files reduces procedure errors during shaping of the root canals, especially during instrumentation of severely curved canals compared with conventional stainless steel hand files.[1] Files separation of NiTi rotary instruments are still major issue during clinical application.[2,3] Also another mechanical deficiency as cyclic fatigue are major complications, cyclic fatigue fractures occurs due to continuous tension and compression repeated cycles at the point of maximum flexure when the file rotates in a severely curved canals.[4]

There are many factors contributed to broken files include instrument size, file cross-sectional, design[5,6], metal surface treatments and the metallurgical characteristics of the Ni-Ti alloy, among others[7,8]. Therefore, attempts were made by several manufacturers for developing of special forms of thermomechanical processing, producing a superelastic NiTi alloy that contains a stable martensitic phase under clinical conditions [9].

Many improvements in new manufacturing techniques as thermal and surface treatment have been developed to improve the mechanical properties, increasing the cyclic fatigue resistance of Ni-Ti rotary instruments.[10] Thermomechanical processes which were applied to conventional Ni-Ti wire improves and change the microstructure of NiTi alloy and therefore extends the files resistance to fracture and the life span. A special newly introduced NiTi alloy termed as M-wire introduced by (Dentsply, Tulsa, Oklahoma, USA) was developed by new advanced manufacturing technique. Its enhancement in flexibility and cyclic fatigue resistance is due to the unique nanocrystalline martensitic microstructure of the treated alloy.[11] M-wire is currently used for the manufacturing of different rotary systems. Another thermal treatment procedure was developed termed R-Phase technology that optimizes the microstructure and mechanical properties also improves the fatigue resistance of the NiTi alloys.[12]

Recently, NiTi rotary instruments made from a NiTi controlled memory wire (CM wire, DS Dental, Johnson City, TN, USA) have been introduced. The manufacturer claims that these instruments have superior flexibility and fatigue resistance 300% than conventional NiTi rotary instruments made from superelastic wire [13].

The M3 Pro Gold Rotary file (United Dental, Shanghai, China) is a newly introduced NiTi instrument that is designed to be used in continuous rotation motion with an inactive tip and a convex triangular cross-section size 25/ 0.04 taper. The manufacturer claims that this instrument allows quick and safe preparation especially in curved root canals due to high flexibility. M3 Pro Gold files are manufactured with a CM wire associated with an advanced triple surface coating, which allows greater flexibility and cyclic fatigue resistance to the file [14].

The AF F One Blue Rotary file (Fanta Dental Material Co., Shanghai, China) is also newly introduced NiTi instrument in the market that is designed to be used in continuous rotation motion with an inactive tip and a flat surface design 25/ 0.04 taper. The manufacturer claims that this instrument allows quick and safe preparation especially in curved root canals due to high flexibility, manufactured with a CM wire associated with a titanium oxide surface treatment, which allows better flexibility, hardness and resistance to fracture[15].

Hyflex EDM Rotary file (Coltene/Whaledent, Cuyahoga Falls, Ohio, USA), a CM wire with advance surface treatment electrical discharged machining technique (EDM) giving the crater like appearance of the surface represented by pitting and dimpling with variable taper and different cross section giving the file greater flexibility, hardness and resistance to cyclic fatigue.[16]

MATERIALS AND METHODS

In this study one hundred and twenty rotary files were used from three different rotary systems with the same thermomechanical treatment – controlled memory wire (CM Wire) and different surface treatment technique using rotation motion. ***Group 1***: M3 Pro Gold (United Dental, Shanghai, China) (CM Wire), ***Group 2***: AF F One Blue (Fanta Dental Material Co., Shanghai, China) (CM-Wire) and ***Group 3***: Hyflex EDM (Coltene/Whaledent, Cuyahoga Falls, Ohio, USA). Each group contained 40 files with (IS0 tip size # 25 and a 0.04 taper). Before applying the cyclic fatigue test, each file was tested for any possible manufacturing defects and deformation with a scanning electron microscope SEM (Quanta FEG 250, Egypt) under magnification of 200X to 3000X.

The cyclic fatigue test was preformed using a custom-made apparatus that was specially designed for this experiment by using a modification of the apparatus described by Larsen *et al*. [8] and Capar *et al*. [9]

A Cyclic fatigue testing block was fabricated as a stainless-steel metal block with dimensions 9.5 cm length x 4cm height and 0.5mm in thickness. Four simulated canals were created in this stainless-steel block (2 with angle 90° radius 2.5mm and 5mm) and (2 with angle 60° radius 2.5mm and 5mm) all files 0.04 taper and tip diameter with 0.2 mm offset for canal width. The depth of the simulated canals was 2mm to accommodate the size and taper of the files thus providing the instrument with a suitable trajectory. A flexiglass cover that prevent fractured part of the file from slipping out was attached to the side of the block. Figure (1a) Four holes were made at the corners of the cyclic fatigue testing block to allow for fixation of the block to the cyclic fatigue testing apparatus. For each of the tested rotary files solid works software (Dassault Systèmes, S. A. Vélizy, France) was used to determine canal geometry that follow the tested file. The angle of curvature was calculated by Pruett's method 4 which is described as the curvature radius for measuring root canal curvatures, this method which defines the canal shape using two parameters: the angle of curvature and the radius of curvature ***Figure (1a)***

Cyclic fatigue testing apparatus is a custom-made stainless-steel apparatus which was designed as described by Gianluca Plotino [17]. The apparatus was made from stainless steel and was composed of a 2 cm thick stainless-steel sheet, which is considered the main part with 60 cm x 60 cm dimensions. In the upper right two parallel slots 40 cm in length were made which are used in fixing the upper sliding stainless sheet of dimensions 10 cm x 10 cm which was considered the movable tray that made positioning the hand-piece easy and reproducible. Fixing the two sheets together was done by the presence of 2 screws on the upper slot while the stainless-steel block was fixed by other 2 screws found on its top surface also found on the movable stainless sheet with diameter 2mm. Also, the hand-piece rested to the main sheet by a non-movable tray which was fixed to, the main sheet by 4 screws and it was stabilized to the non-movable sheet by two cable ties passing through 4 slots**.** ***Figure (1b)***

Cyclic fatigue testing after the hand piece was mounted on the apparatus, then each instrument according to each group and subgroup was precisely positioned to the block. Instruments were rotated until fracture occurs and time taken for fracture was recorded. The instruments were rotated at torque and speed according to manufacture instruction of files using a 16:1 reduction hand piece (ENDO-MATE TC2 Reduction Heads from NSK, Japan) powered by a torque-controlled electric motor (ENDO-MATE TC2 Motor from NSK, Japan). A synthetic oil (Pana spray plus from NSK, Japan) filled all the stainless-steel canal space was used to reduce the friction of the file with block before testing every file. A flexiglass top cover was used to allow visualization of the file while rotating in the canal and the fracture of the instrument it also helped in maintaining the oil in the canal for a longer period and preventing the file from deviating out of the canal space.

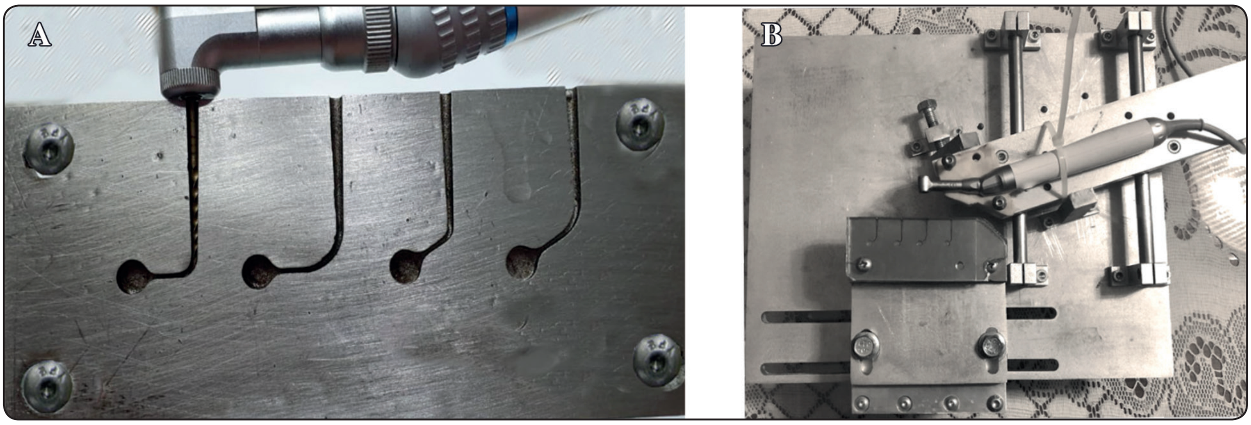

Fig. (1): Cyclic fatigue testing block (a); Cyclic fatigue testing apparatus(b)

Method of evaluation:

A- Number of Cycles to Failure (NCF): Static cyclic fatigue testing was performed for each file at 500 rpm and torque 2.5N/cm for 3M pro Gold, AF Fone Blue and Hyflex EDM with continuous rotation. Working length was standardized to 16 mm for each file. During the testing, engine oil was used to reduce the friction between files and metal block. All files were rotated until fracture occurred and the time to fracture was recorded in seconds. The length of the fractured file tip was also measured with a digital micro caliper (Aydal, Istanbul, Turkey) and recorded. Instrument fracture was visually and audibly detected and the time until fracture was recorded in seconds by using a digital stopwatch manually operated, started at the moment the motor was turned on, and stopped at fracture detection. All fatigue tests were performed by the same operator the number of cycles to fracture (NCF) was then calculated using the simple formula: ***Number of rotations = (recommended speed per minute x time to fracture in minutes)*** [18]

B-Mode of failure: The fractured segments were collected and ultrasonically cleaned in an ultrasonic bath using absolute alcohol for 90 seconds. To remove the debris and the oil remnants from the surface of the file before the examination to determine the fracture mode, random samples were chosen to be tested under scanning electron microscope (SEM) (Quanta FEG 250, Egypt). Samples were mounted vertically then horizontally to obtain top view and lateral view respectively using various magnifications ranging from 200x to 3000x:

1- Top views with high magnification were taken to evaluate the details and patterns of cyclic fatigue fracture.

2- Lateral views were taken to evaluate signs of plastic deformation (if any), disruptions of cutting edge and manufacturing grooves and quality of surface treatment of the files of the three groups. [19]

The instruments were investigated under SEM for topographic features of the separated part of the file that all are explained in ***Table 1.***

Statistical analysis:

Statistical analysis was performed by Microsoft office 365 (Excel) and Statistical Package for Social Science (SPSS) version 20. Means and standard deviations of (NCF) number of cycles till fracture were calculated for each group. The data were analyzed by variables ANOVA two-way analysis of variance and Tukey's test was performed at the 95% confidence level ($P = 0.05$). All statistical analyses were performed using SPSS software (SPSS Inc, Chicago, Illinois, USA).

TABLE (1) Patterns and signs of cyclic fatigue fracture.

Defect	Definition
Metallic strips	Visible flashes of metal protruding irregularly from the surface
Metallic folds	Rolled over metallic dim appearing along the cutting edge
Disruption of the cutting edges	The loss of the regular continuous shape of the blades
Micro fractures	A surface defect that appears as surface fissuring and discontinuity, it appears mainly as accentuation of machine grooving.
Cracks	Break down without complete separation of parts
Fractures	Complete separation of parts
Scrapings	Removed areas from surface which may be due to excessive frictional force
Pitting	Small, excavated and punched out areas on the surface
Craters	Large excavated areas on the surface
Corrosion	Disintegration of the NiTi alloy due to chemical reaction appears darker in the color than the rest of the surface
Fretting	Observable corrosion with friable surface texture
Debris	Small particles made of materials removed from the canal

RESULTS

Effect of file type, angle of curvature and radius on Number of cycles to failure (NCF):

The mean values and standard deviations of Number of Cycles to Failure (NCF) of each group were calculated and represented in ***Table (2,3).*** *At angle 60 degree;* there were no significant differences between the three groups in 5mm radius ($P< 0.05$) while in 2.5mm radius there were no significant differences between the M3 Pro Gold group and the AF F One Blue group ($P < 0.05$) and there were significant differences between the Hyflex EDM and the other two groups ($P >0.05$). *At angle 90 degree;* there were no significant differences between M3 Pro Gold and AF F One Blue rotary file in 5mm radius ($P < 0.05$) and significant differences between the Hyflex EDM and the other two groups ($P >0.05$) whilst in 2.5mm radius there were no significant differences between M3 Pro Gold and AF F One Blue rotary file ($P < 0.05$) and highly significant differences between the Hyflex EDM and the other two groups ($P >0.001$) ***Figure 2***.

Table (2) Mean values (±standard deviation) for number of cycles to fracture of each group

Angle / Radius	Radius	M3 Pro Gold	AF F one Blue	Hyflex EDM
60 Degree Angle	2.5 mm	6981.75 ± 421.6	6774.61 ± 985.82	7982.41 ± 396.63
	5 mm	7636.93 ± 716.72	7526.89 ± 661.99	8359.21 ± 880.72
90 Degree Angle	2.5 mm	134.82± 40.403	207.99 ± 154.87	4558.8 ± 1499.9
	5 mm	4095.9 ± 1175	3504.9 ± 1108.1	5294.2 ± 929.72

TABLE (3) The significant differences between all groups at angles 60 and 90 and radii 2.5mm and 5mm

	Angle 60 degree		Angle 90 degree	
	2.5mm Radius	5mm Radius	2.5mm Radius	5mm Radius
M3 Pro Gold vs AF F one Blue	ns P>0.05	ns P>0.05	ns P>0.05	ns P>0.05
M3 Pro Gold vs Hyflex	*P<0.05	ns P>0.05	**P<0.001	*P<0.05
AF F one Blue vs Hyflex	*P<0.05	ns P>0.05	**P<0.001	*P<0.05

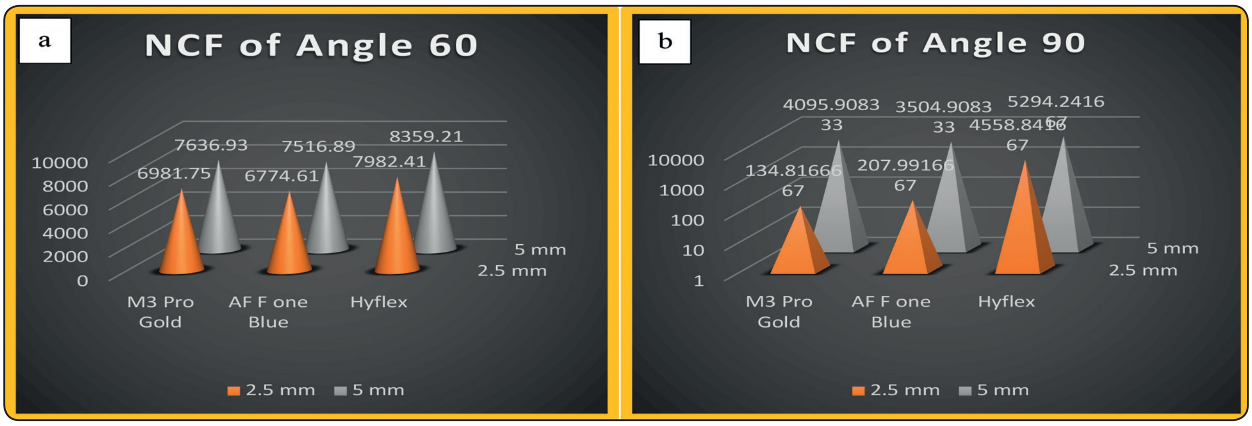

Fig. (2) Bar chart showing the effect of different radii of curvature on NCF in 60 degree angle (a) and 90 degree angle (b) using the three types of rotary files

II-Results of Scanning Electron Microscope:

Scanning Electron Microscope images cross section view of a samples of the fracture surface of M3 Pro Gold, AF F one Blue and Hyflex EDM at 800X and 1600X magnification showed similar and typical features of cyclic fatigue pattern which have high resemblance to ductile fracture (ability of the material to undergo plastic deformation before fracture); where micro voids produced within the metal, nucleation growth and micro voids coalescence which ultimately weaken the metal result in fracture, concentric abrasion marks and fibrous dimple marks at the center of rotation was observed. Plastic deformation seen because of slip; the process by which a dislocation moves in response to shear stresses, also contributes to ductile fracture. are shown in ***Figures (3)***

Scanning electron microscopy longitudinal view of sample of each group at 200X and 3000X magnification showed the different surfaces treatment of the rotary files; the titanium oxide coating of AF F One Blue which shows continuous homogenous coating with disruption of the cutting edges surface and M3 Pro Gold coated with advanced surface treatment technique showed disruption of the cutting edges surface. Hyflex EDM showed a crater like surface with pitting and dimpling surface. ***Figure 4***

Cross section view

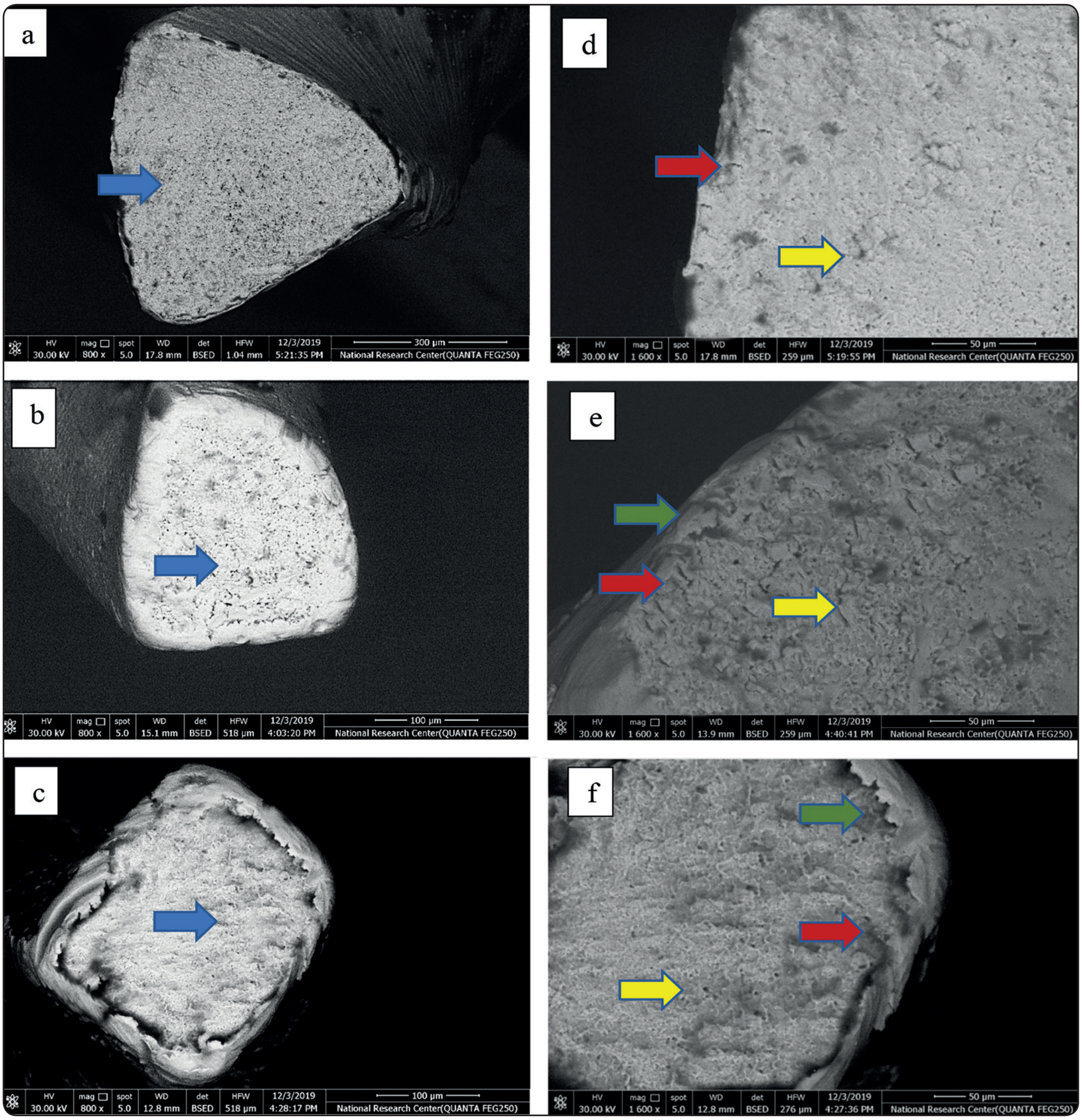

Fig. (3) SEM image for Top view of fractured M3 Pro Gold (a,d), AF F one Blue (b,e) and Hyflex EDM (c,f) rotary files at magnification of 800X and 1600X showing the different cross sections of the files, the fibrous appearance indicating ductile fracture (blue arrow), metallic folds as rolling of the edges (green arrow) and pitting yellow arrow, the surface microcracking (Red arrow)

Longitudinal view

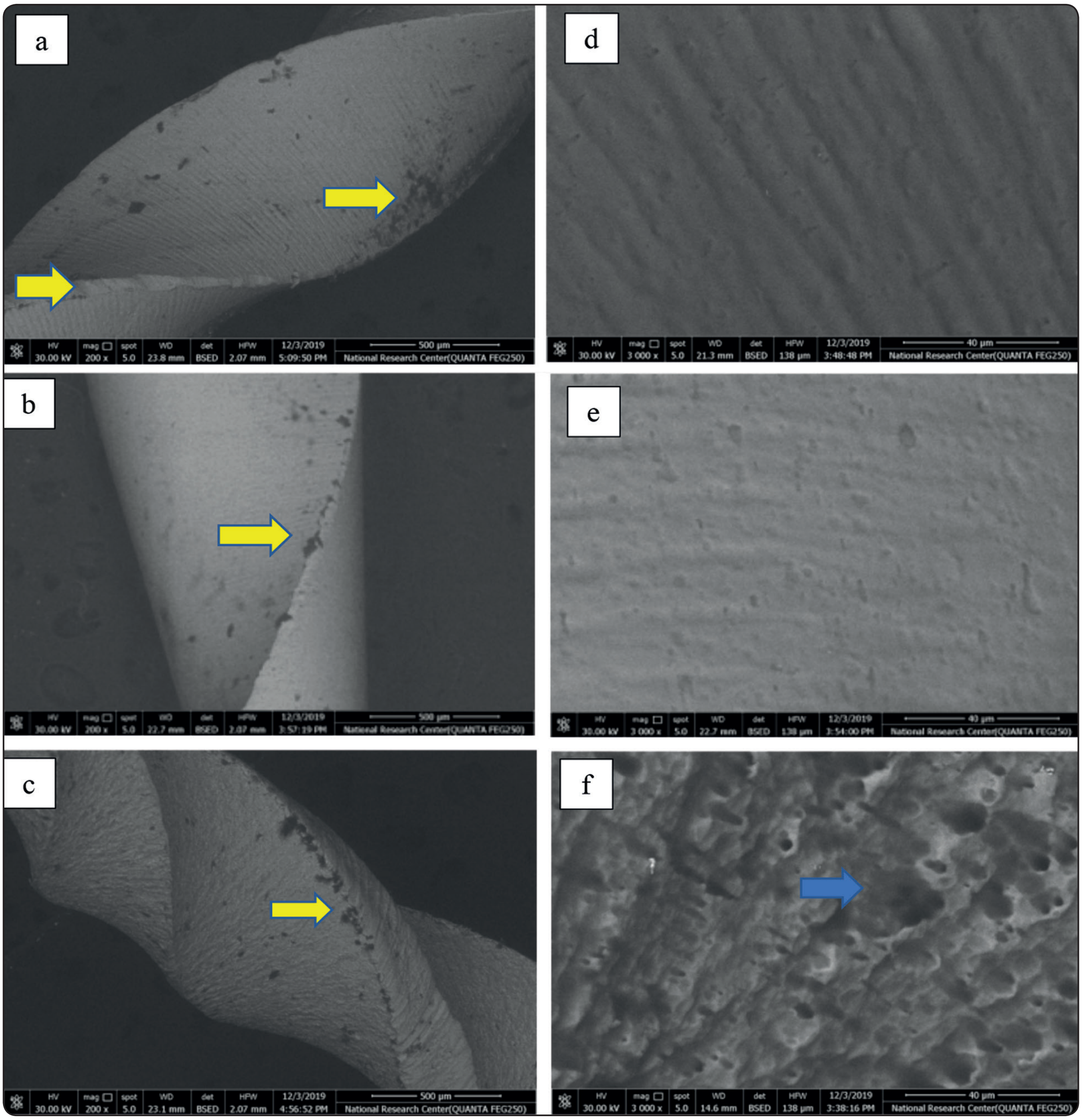

Fig. (4) SEM image for longitudinal view for the surface treatment of fractured M3 Pro Gold (a,d), AF F one Blue (b,e) and Hyflex EDM (c,f) rotary files at magnification of 200X and 3000X showing multiple pitting and dimpling (blue arrow), disruption of the cutting edges and surface (yellow arrow)

DISCUSSION

New generation of rotary files are fabricated from thermo-mechanically treated alloys have enhanced the resistance to cyclic fatigue and increase flexibility in addition to surface treatment that enhance the hardness of the files and decreases tendency file fracture. Factors influencing the fatigue resistance include file design, cross-sectional geometry and diameters of core, tip size, and taper of the tested file. [20,21] Furthermore, radii and degree of curvature, rotation speed, torque, and movement kinematics (continuous, reciprocal, or adaptive) also perform an important role in the cyclic fatigue resistance of files. [22]

Gao Yet al. [23] mentioned in his study that the speed and type of motion (reciprocal or rotational) of files may also affect the cyclic fatigue resistance. For these reasons, standardization of these variables is a must. Although, several studies compared the cyclic fatigue resistance of different NiTi rotary files, with different cross section, degree of taper, or different sizes. [4,6,8,21] By this way, only the influence of the difference of manufacturer method and the surface treatment of the files on cyclic fatigue could be compared.

In this present study, standardized of artificial canals (60° and 90° angle of curvature and a curvature radius of 2.5mm and 5mm) and same torque, rotation speed (500 rpm), rotation type (continuous) were set to minimize the influence of variables and also all groups were CM-wire different manufacturing methods influenced the fatigue resistance of the endodontic files produced by different companies and different surface treatment. ***Shen et al.*** [13,24] observed increased fatigue resistance in files manufactured via proprietary CM wire processing. The superiority of CM files in terms of cyclic fatigue resistance was also proven in previous studies. [4, 25, 26] The reason for this may be that for a given strain, a more flexible file would experience less stress, allowing for a longer fatigue lifetime [27]. Cyclic fatigue behavior of the files using time to fracture data was compared. Similar to the present assessment method, previous researchers used number of cycles to fracture for the failure of files [8, 9, 7,10, 20]

The AF F One Blue Rotary files, M3 Pro Gold and Hyflex EDM rotary files were compared because they have the same thermomechanical treatment in their manufacturing technique (CM wire) but differ in surface treatment and design and geometrical features. Sizes 25 with 0.04 taper of instruments were tested because these sizes are commonly used during instrumentation. [21]

According to results, when same size of the instruments was compared, the Hyflex EDM files exhibited higher cyclic fatigue resistance and angular rotation to fracture than M3 Pro Gold and AF F One Blue Rotary files especially at angle 90 degree radius 2.5mm, while no significant difference was observed between M3 Pro Gold and AF F One Blue Rotary files at angle 60 and 90 degree with both radius. These results are probably due to the different NiTi alloys treatment temperature and the differences in surface coating and treatment [28, 29].

Pedullà E et al.[30] conducted DSC analysis to M3 Pro Gold files, as CM wire, which showed a mixture of martensite and austenite at room temperature. In particular, M3 Pro Gold files were measured to be 36.39°C, at their heating curves. This means that at human body temperature (around 37°C) [31], the M3 Pro Gold files are in a mixed austenite and martensite phase. Since martensite is less stiff than austenite and Martensite is more likely to deform than austenite because it has a twinning process, which refers to an internal movement of lattices without breaking atomic bonds by absorbing stress. [32]

The AF F one Blue files were introduced to produce NiTi instruments by an innovative methodology (a complex heating and cooling treatment) that uses a unique process that controls the material's memory similarly to the shape memory NiTi rotary files.

HyFlex EDM rotary files, mainly composed of martensite and R phase, revealed special structural properties, such as increased phase transformation temperatures and higher hardness when compared with other manufactured CM wire instruments. The increase in the plasticity of thermally treated files is thought to be due to the increase of the proportions of the R-phase and martensite. The R-phase has the lowest shear modulus among the 3 phases [33]. The different phase composition and the improved hardness may shed light on the enhanced mechanical behavior of electro-discharge machined instruments.[34] These findings with (*Gündoğar M and Özyürek T)*

Anderson et al. [35] concluded that electro-polishing surfacing is likely reduce surface irregularities that serve as points of stress concentration and crack initiation. Manufacturing process may serve as nucleating sites for the micro-voids and crack propagation at grain boundaries and surfaces leading to instrument fractures during clinical use ***Alapati et al.*** [36] The high density of surface defects facilitates the crack nucleation stage and the fatigue failure is largely a crack propagation process ***Kuhn et al.*** [37]. Thus, it is possible that the multitude of machining marks on the surface of a ground instrument would lead to crack initiation at multiple locations in which the resolved shear stress is greater than that required for crystallographic slip to occur ***Kim et al.*** [38]

Under the conditions of this study, the surface treated instruments may improve cyclic fatigue resistance while maintaining the torsional resistances and mechanical properties, this within agreement ***Kim et al.***[39] ***and Plotino G et al*** [40]***.***

CONCLUSIONS

All new thermomechanical treatment technique - controlled memory CM-Wire rotary systems represented great performance and remarkable enhancement in cyclic fatigue resistance but the Hyflex EDM rotary files had the highest cyclic fatigue resistance among the M3 Pro Gold and AF F one Blue rotary files. Also surface treatment of the rotary files enhancing the cyclic fatigue resistance by decreases surface micro cracks and the tendency to fracture.

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