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Date: **25 MAY 2017**

Torsional stress, cyclic fatigue, and thermal analyses of heat-treated and conventional nickel-titanium rotary instruments

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Objective: Compare phase-transformation temperatures, torsional-stress properties, and effect of rotational speed per minute (RPM) on cyclic-fatigue life of two proprietarily heat-treated nickel-titanium (NiTi) rotary files (EdgeSequel Sapphire® [ESS] and Vortex Blue® [VB]) to conventional NiTi rotary files (ProFile® [P]).
Materials and Methods: Torsional moments (TMs) and rotational degrees (RDs) of the files (size=25/0.04, n=20/brand) were measured using a custom-built torsiometer. Effect of RPM on file cyclic-fatigue life was assessed using a custom-built 3-peg jig that constrained files to a simulated curvature (90°, r=3.5mm). Files subjected to various RPMs (100, 200, 300, 400, 500, and 600) in air. Measured number of revolutions to fracture (N) per RPM. The phase-transformation temperatures investigated (n=5/brand) by differential scanning calorimeter (DSC). Data analyzed with nonlinear regression and ANOVA/Tukey (α=0.05).
Results: TMs (g-cm) and RDs (°) rankings were: VB (75±8) > P (72±13) > ESS (58±5) and ESS (793±122) > VB (515±38) & P (515±63), respectively. DSC analyses showed ESS (33±6°C) and VB (32±4°C) have austenite finish temperature (Af) below body temperature, P (47±4°C) has Af above body temperature. For ESS and VB, RPM correlated with N, where RPM-N curves were best fitted by power regression, r2=0.987 and 0.969 respectively. For P, RPM-N power regression was weakly correlated (r2=0.116).
Conclusion: Irrespective of thermal history, files with high TMs conveyed low RDs. Heat-treated files displayed higher cyclic-fatigue resistance than conventional files under similar 100-600 RPM range and have an endurance limit of RPM=100, below which N was theoretically infinite. RPM appeared independent of N for conventional files.

Introduction:

Successful endodontic treatment depends not only on a series of sequential cleaning and shaping of the root canal system but also relies on the manner in which files fatigue or fail during mechanical endodontic instrumentation. NiTi alloy has been in use in rotary endodontics for several years, due to its super-elastic properties.[1] The metal displays two principle phases an austentite and a martensite phase. Austenite phase is characterized by increased toughness. The martensite phase is characterized by increased flexibility.[2] Differential scanning calorimetry elaborates the temperature required for NiTi phase changes.[3]

Historical processes of optimizing the NiTi attributes include electro-polishing treatment, passivation, and modification of NiTi metallurgic composition. The current strategy in manufacturing endodontic files is to use thermo-mechanical processes to minimize or remove impurities and defects, resulting in strength and flexibility, which could potentially improve both cutting efficiency and fatigue resistance. [4]

Two major stresses that shorten life of endodontic files are torsional fatigue and cyclic fatigue. Torsional failure is defined as rotary file failure due to binding of a portion of the file while the engine attempts rotation.[5 6 7 8] Fatigue is defined as transitional weakening of a material due to cyclic loading and unloading characterized by fracture below its ultimate tensile strength. [9]

The objective of this study is to compare phase-transformation temperatures, torsional-stress properties, and effect of rotational speed per minute (RPM) on cyclic-fatigue life of two proprietarily heat-treated nickel-titanium (NiTi) rotary files (EdgeSequel Sapphire® [ESS] and Vortex Blue® [VB]) to conventional NiTi rotary files (ProFile® [P]).

Null hypothesis-1: Heat-treated NiTi instruments (ESS and VB) will not demonstrate an increased ability to withstand torsional stress and cyclic fatigue when compared to conventional NiTi instruments (P).
Null hypothesis-2: RPM will not affect the cyclic fatigue resistance of all groups.

Materials and methods:

Files from three brands, P, VB, and ESS were subjected to differential scanning calorimetry, torsional fatigue testing, and cyclic fatigue testing. All files tested were of the same dimensions, 25mm length, ISO size 25, and taper 0.04.

Torsional fatigue testing
A total of 45 files were tested, Experimental groups were assigned according to the following groups: Profile n=15, Vortex Blue n=15, and EdgeSequel Sapphire n=15. See table 1.
Torques (Ts) and rotational degrees (RDs) of the files (size = 25/0.04, n = 15/brand) were measured at room temperature (23 ± 2 ^{o}C) using a custom-built torsiometer (Sabri Dental Enterprises) in accordance with ISO 3630-1. The device features a removable chuck and a fixed chuck. The file tip was inserted 3mm, as measured by a custom guide, into the removable chuck. The handle of the file was inserted into the fixed chuck. The fixed chuck rotated a torsional rate of 2 rotations per minute in the clockwise direction. The torsiometer instrument provided continuous readings of torsional moments and rotational degrees until file separation occurred. Following separation, files were measured to determine site of separation with a digital caliper. Data was normalized to the 3mm location on the file.

Cyclic fatigue limit testing

A total of 360 files were subjected to cyclic fatigue fracture. 120 files per brand were subdivided into groups based on RPM. A total of 18 subgroups were created. For each subgroup, n=20. P100 n=20, P200 n=20 etc.

Cyclic fatigue study was completed using a custom-built adjustable pegboard model; adapted from a description by Cheung in 2007.[10] The deflection angle was set to 90°. Radius of curvature was 5mm, as described by Nguyen[11]. A Promark motor was used to control RPM and torque. The torque setting for all tests was 520 g-cm.
Files were cycled until fracture at room temperature (23 ± 2 ^{o}C), under a constant drip of synthetic oil (WD-40). Time to failure was multiplied by the RPM to determine the number of cycles to failure (N). The mean N of each subgroup were compared across brands groups and RPM subgroups.

Differential Scanning Calorimetry
A 3-4 mm sample was sectioned from each file tip (n = 5/brand) to investigate the phase-transformation temperatures by DSC (Model 823^{e}, Mettler Toledo) in “as-received” condition. Samples were cut from each instrument using manual diagonal cutting and weighed to an accuracy of 0.01 mg before being placed in a preweighed Tzero aluminum pan (TA Instruments, New Caste, DE) and sealed. Samples were a single piece removed from the apex of the file.
Each sample was placed in a Mettler Toledo DSC 823 along with an empty Tzero aluminum reference pan. With liquid nitrogen as coolant and nitrogen gas for purging, at 5 ^{o}C/minute, the samples were first heated to 100 C and then cooled to -60 C at a rate of 5 C/ min followed immediately by a heating cycle at 5 C/min up to 100 C. The heating/cooling cycle was performed 3 times per sample. The DSC plots were analyzed by the DSC manufacturer software. The starting and finishing temperatures were determined as the intersection of the line tangent to the curve at its point of inflection and the baseline.
Statistics: Data from all tests were analyzed with nonlinear regression and ANOVA/Tukey (α=0.05).

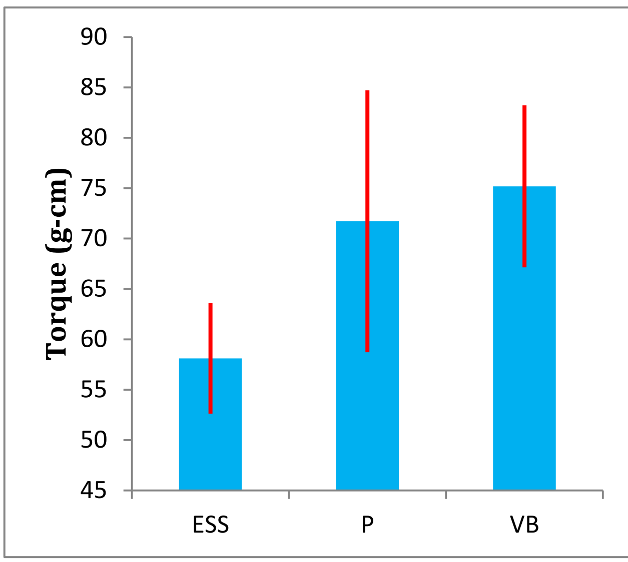
Results:

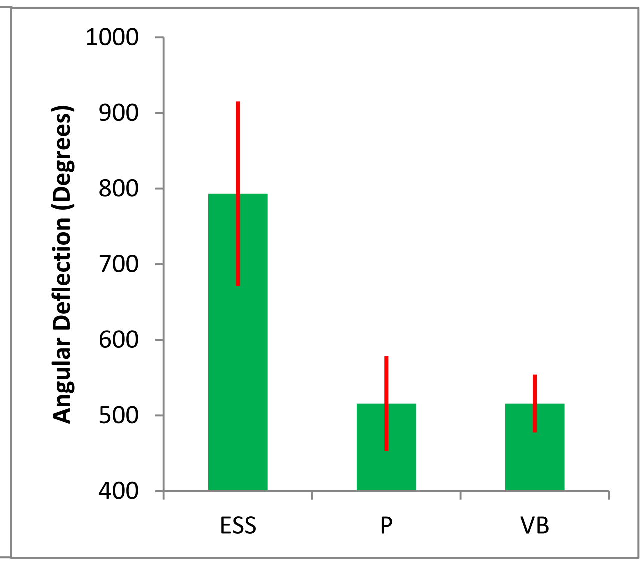
Torsional fatigue results
Mean torque values were significantly lower for ESS compared with P and VB.
Torsional moments VB: 75+- 8, P (72+-13), ESS 58+-5. Rotational degree values were significantly greater for ESS compared with P and VB. Rotational degrees ESS 793+-122, VB 515+-38, P 515+-63. See table 2 for values.

Table 2

	ESS		VB		P	
	Peak Torque	Peak Angle	Peak Torque	Peak Angle	Peak Torque	Peak Angle
Test	(g-cm)	(Degrees)	(g-cm)	(Degrees)	(g-cm)	(Degrees)
1	59	723	77	454	79	485
2	83	1001	69	519	81	565
3	60	795	86	507	109	673
4	59	948	80	526	87	494

5	53	781	88	496	56	480
6	60	987	76	493	90	583
7	54	634	71	566	75	445
8	60	811	64	511	69	488
9	53	729	99	540	52	546
10	60	830	59	544	49	458
11	57	794	73	477	75	488
12	52	682	76	483	81	506
13	59	873	73	481	57	556
14	62	729	65	537	64	452
15	50	581	72	602	73	579



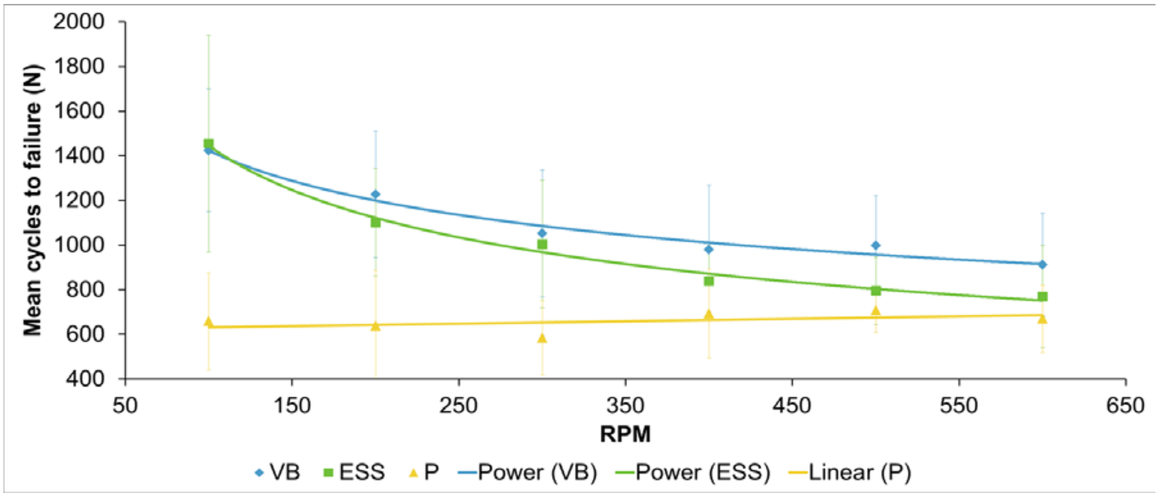


Cyclic fatigue testing results.
N is inversely proportional to RPM for ESS and VB but not for P. Generally, increasing RPM decreases the number of cycles that leads to separation. [12]

See Table 3 for values.
Table 3

ESS			VB			P		
RPM	Mean N	SD	RPM	Mean N	SD	RPM	Mean N	SD
100	1454	495.19	100	1424	276.89	100	658	223.97
200	1101	240.33	200	1227	284.25	200	638	247.11
300	1004	285.69	300	1052	285.42	300	584	165.98

400	839	160.86	400	980	288.14	400	693	199.42
500	795	151.04	500	998	221.62	500	708	100.2
600	769	228.34	600	911	230.62	600	670	150.67

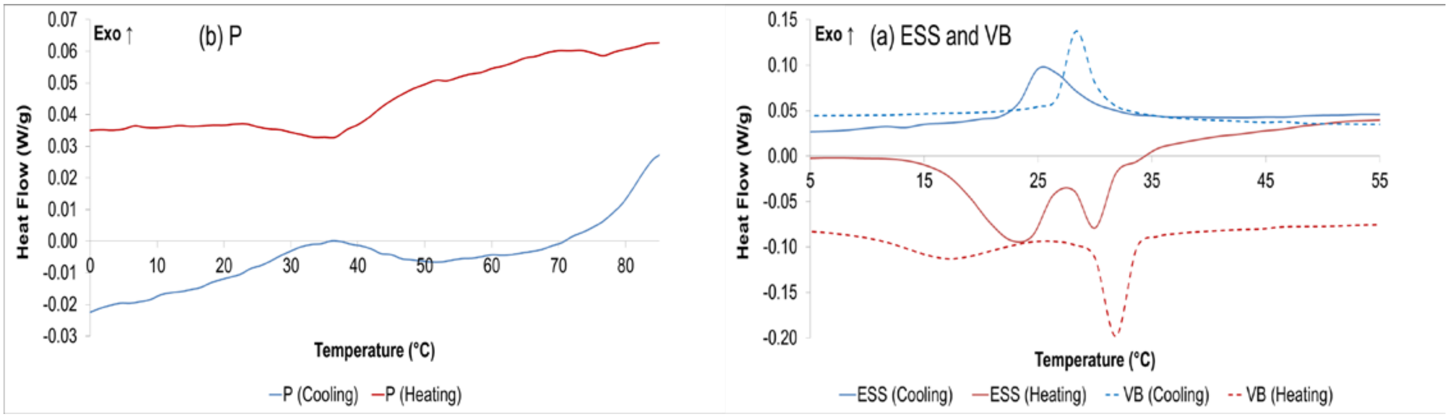


DSC-
Average phase change temperatures of Profile instruments (As, Af vs. Ms, Mf) were relatively lower compared to Edge and Vortex blue. Heat-treated files frequently showed an intermediate phase change. The intermediate phase is called the R-Phase, where properties of both phases area present.[13]
Hysteresis temperature is defined as the difference between the temperatures at which the material is 50% transformed to A phase upon heating and 50% transformed back to M phase upon cooling.
See table 1 for values.

Table 1

Temperature	ESS		VB		P	
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**
A-start (°C)	**24.2**	**5.9**	**27.6**	**5.4**	**21.8**	**5.0**
A-peak (°C)	**29.4**	**5.0**	**30.0**	**4.1**	**36.4**	**0.7**
A-finish (°C)	**33.2**	**6.5**	**32.4**	**3.5**	**47.2**	**3.7**
M-start (°C)	**33.1**	**4.0**	**31.2**	**0.3**	**44.8**	**3.9**
M-Peak (°C)	**27.9**	**2.6**	**28.5**	**0.8**	**33.9**	**2.3**
M-finish (°C)	**23.2**	**6.7**	**26.7**	**0.8**	**20.3**	**5.3**
Hysteresis temperature (°C)	**1.5**		**1.5**		**2.5**	
A-finish to M-finish range (°C)	**10.0**		**5.7**		**26.9**	

Representative DSC Curves



Discussion:

The null hypothesis-1 was rejected for ESS cyclic and torsional fatigue, while VB was rejected for cyclic fatigue only. The null hypothesis 2 was rejected for VB and ESS. Even though ESS[14] and VB[15] have both been heat-treated, the ESS system was able to tolerate significantly more rotational degree than VB under torsional stress. This ability may have a clinical advantage since having an improved rotational-degree tolerance tends to increase the file's unwinding capacity to withstand high torsional stress as well as cyclic fatigue at lower stress levels (i.e. decreased RPM).

For ESS and VB, the martensitic-to-austenitic phase transformation occurs over a narrow temperature range in which the shape-memory property of these NiTi alloys recovers the form it had prior to its deformation and consequently acquires superior elasticity. Furthermore, the ESS and VB hysteresis temperatures, vacillating between room and oral temperature, are more ideal than P for clinical operating conditions since any deformation that the ESS and VB NiTi-alloys had introduced at room temperature can be easily erased on the application of oral temperature, which is high enough to ensure complete transformation to the austenite phase. However, the austenitic-finish temperature at 47.2^{o}C for P is much greater than oral temperature and therefore is neither clinically conducive nor workable. At room temperature, all three systems (ESS, VB, and P) are equally sensitive to torsional stress since they are mostly in their martensitic phase, which has a lower density and less rigidity than in their austenitic phase.

A criticism of the study design was that it was not conducted at the same temperature as the oral environment. de Vasconcelos et al[16] recommended conducting fatigue tests at the oral temperature. Additionally, the DSC sample sizes were relatively small when compared to similar studies.

Conclusion:

Irrespective of thermal history, files with high TMs conveyed low RDs. Heat-treated files displayed higher cyclic-fatigue resistance than conventional files under similar 100-600 RPM range and have an endurance limit of RPM=100, below which N was theoretically infinite. RPM appeared independent of N for conventional files.

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Torsional fatigue testing
A total of 45 files were tested, Experimental groups were assigned according to the following groups: Profile n=15, Vortex Blue n=15, and EdgeSequel Sapphire n=15. See table 1.
Torques (Ts) and rotational degrees (RDs) of the files (size = 25/0.04, n = 15/brand) were measured at room temperature (23 ± 2 ^{o}C) using a custom-built torsiometer (Sabri Dental Enterprises) in accordance with ISO 3630-1. The device features a removable chuck and a fixed chuck. The file tip was inserted 3mm, as measured by a custom guide, into the removable chuck. The handle of the file was inserted into the fixed chuck. The fixed chuck rotated a torsional rate of 2 rotations per minute in the clockwise direction. The torsiometer instrument provided continuous readings of torsional moments and rotational degrees until file separation occurred. Following separation, files were measured to determine site of separation with a digital caliper. Data was normalized to the 3mm location on the file.

Cyclic fatigue limit testing

A total of 360 files were subjected to cyclic fatigue fracture. 120 files per brand were subdivided into groups based on RPM. A total of 18 subgroups were created. For each subgroup, n=20. P100 n=20, P200 n=20 etc.

Cyclic fatigue study was completed using a custom-built adjustable pegboard model; adapted from a description by Cheung in 2007.[10] The deflection angle was set to 90°. Radius of curvature was 5mm, as described by Nguyen[11]. A Promark motor was used to control RPM and torque. The torque setting for all tests was 520 g-cm.
Files were cycled until fracture at room temperature (23 ± 2 ^{o}C), under a constant drip of synthetic oil (WD-40). Time to failure was multiplied by the RPM to determine the number of cycles to failure (N). The mean N of each subgroup were compared across brands groups and RPM subgroups.

Differential Scanning Calorimetry
A 3-4 mm sample was sectioned from each file tip (n = 5/brand) to investigate the phase-transformation temperatures by DSC (Model 823^{e}, Mettler Toledo) in "as-received" condition. Samples were cut from each instrument using manual diagonal cutting and weighed to an accuracy of 0.01 mg before being placed in a preweighed Tzero aluminum pan (TA Instruments, New Caste, DE) and sealed. Samples were a single piece removed from the apex of the file.
Each sample was placed in a Mettler Toledo DSC 823 along with an empty Tzero aluminum reference pan. With liquid nitrogen as coolant and nitrogen gas for purging, at 5 ^{o}C/minute, the samples were first heated to 100 C and then cooled to -60 C at a rate of 5 C/ min followed immediately by a heating cycle at 5 C/min up to 100 C. The heating/cooling cycle was performed 3 times per sample. The DSC plots were analyzed by the DSC manufacturer software. The starting and finishing temperatures were determined as the intersection of the line tangent to the curve at its point of inflection and the baseline.
Statistics: Data from all tests were analyzed with nonlinear regression and ANOVA/Tukey (α=0.05).

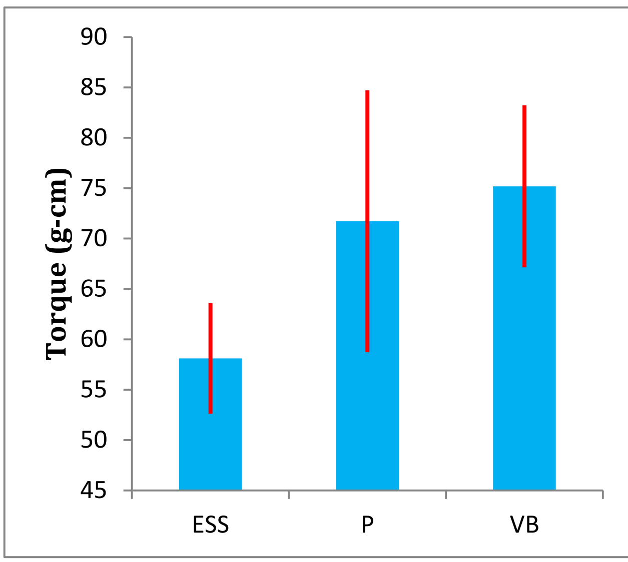
Results:

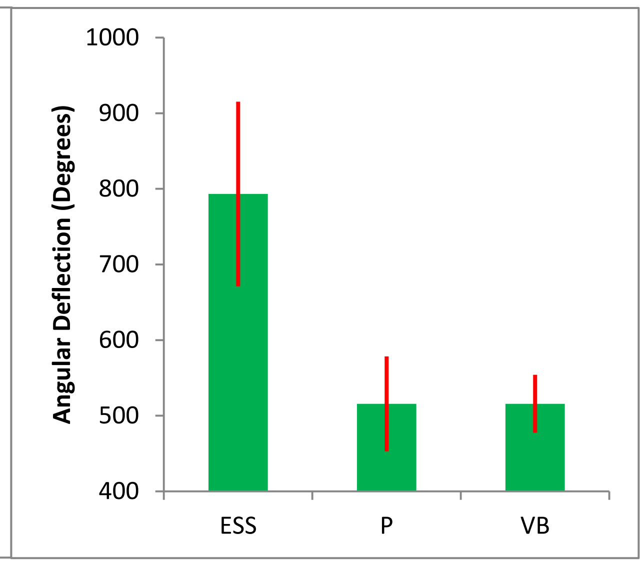
Torsional fatigue results
Mean torque values were significantly lower for ESS compared with P and VB.
Torsional moments VB: 75+- 8, P (72+-13), ESS 58+-5. Rotational degree values were significantly greater for ESS compared with P and VB. Rotational degrees ESS 793+-122, VB 515+-38, P 515+-63. See table 2 for values.

Table 2

	ESS		VB		P	
	Peak Torque	Peak Angle	Peak Torque	Peak Angle	Peak Torque	Peak Angle
Test	(g-cm)	(Degrees)	(g-cm)	(Degrees)	(g-cm)	(Degrees)
1	59	723	77	454	79	485
2	83	1001	69	519	81	565
3	60	795	86	507	109	673
4	59	948	80	526	87	494

5	53	781	88	496	56	480
6	60	987	76	493	90	583
7	54	634	71	566	75	445
8	60	811	64	511	69	488
9	53	729	99	540	52	546
10	60	830	59	544	49	458
11	57	794	73	477	75	488
12	52	682	76	483	81	506
13	59	873	73	481	57	556
14	62	729	65	537	64	452
15	50	581	72	602	73	579



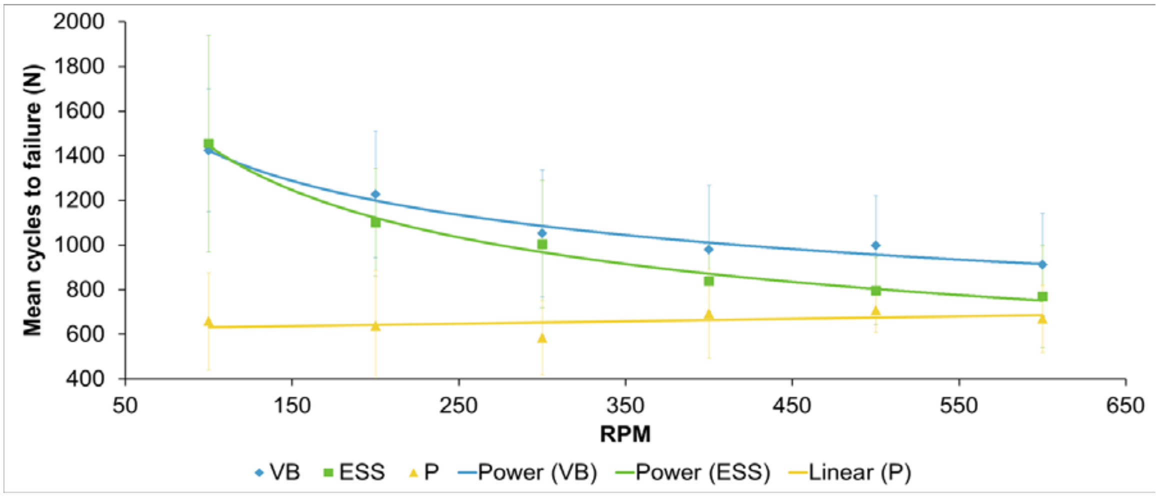


Cyclic fatigue testing results.
N is inversely proportional to RPM for ESS and VB but not for P. Generally, increasing RPM decreases the number of cycles that leads to separation. [12]

See Table 3 for values.
Table 3

ESS			VB			P		
RPM	Mean N	SD	RPM	Mean N	SD	RPM	Mean N	SD
100	1454	495.19	100	1424	276.89	100	658	223.97
200	1101	240.33	200	1227	284.25	200	638	247.11
300	1004	285.69	300	1052	285.42	300	584	165.98

400	839	160.86	400	980	288.14	400	693	199.42
500	795	151.04	500	998	221.62	500	708	100.2
600	769	228.34	600	911	230.62	600	670	150.67

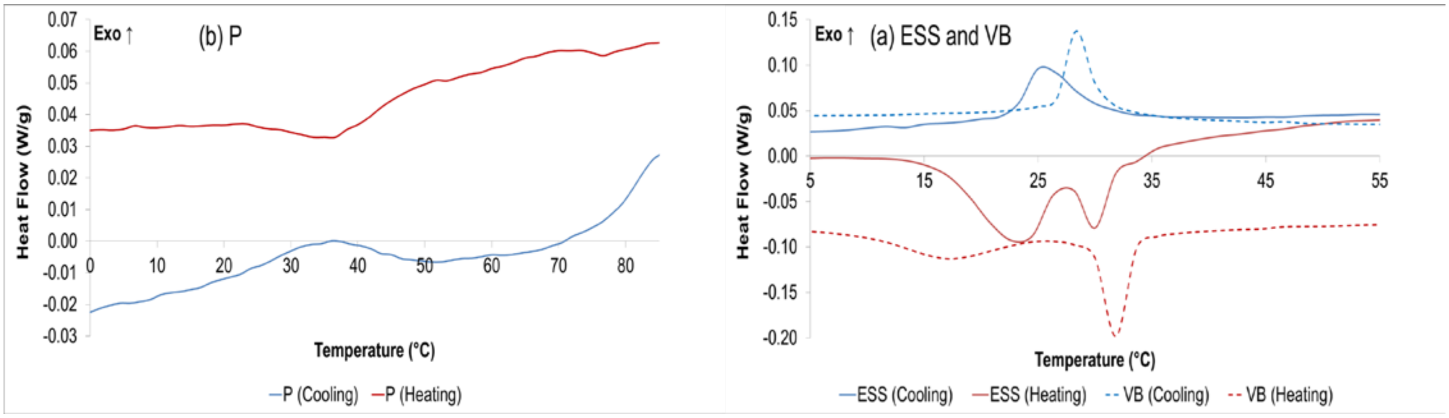


DSC-
Average phase change temperatures of Profile instruments (As, Af vs. Ms, Mf) were relatively lower compared to Edge and Vortex blue. Heat-treated files frequently showed an intermediate phase change. The intermediate phase is called the R-Phase, where properties of both phases area present.[13]
Hysteresis temperature is defined as the difference between the temperatures at which the material is 50% transformed to A phase upon heating and 50% transformed back to M phase upon cooling.
See table 1 for values.

Table 1

Temperature	ESS		VB		P	
	Mean	SD	Mean	SD	Mean	SD
A-start (°C)	**24.2**	**5.9**	**27.6**	**5.4**	**21.8**	**5.0**
A-peak (°C)	**29.4**	**5.0**	**30.0**	**4.1**	**36.4**	**0.7**
A-finish (°C)	**33.2**	**6.5**	**32.4**	**3.5**	**47.2**	**3.7**
M-start (°C)	**33.1**	**4.0**	**31.2**	**0.3**	**44.8**	**3.9**
M-Peak (°C)	**27.9**	**2.6**	**28.5**	**0.8**	**33.9**	**2.3**
M-finish (°C)	**23.2**	**6.7**	**26.7**	**0.8**	**20.3**	**5.3**
Hysteresis temperature (°C)	**1.5**		**1.5**		**2.5**	
A-finish to M-finish range (°C)	**10.0**		**5.7**		**26.9**	

Representative DSC Curves



Discussion:

The null hypothesis-1 was rejected for ESS cyclic and torsional fatigue, while VB was rejected for cyclic fatigue only. The null hypothesis 2 was rejected for VB and ESS. Even though ESS[14] and VB[15] have both been heat-treated, the ESS system was able to tolerate significantly more rotational degree than VB under torsional stress. This ability may have a clinical advantage since having an improved rotational-degree tolerance tends to increase the file's unwinding capacity to withstand high torsional stress as well as cyclic fatigue at lower stress levels (i.e. decreased RPM).

For ESS and VB, the martensitic-to-austenitic phase transformation occurs over a narrow temperature range in which the shape-memory property of these NiTi alloys recovers the form it had prior to its deformation and consequently acquires superior elasticity. Furthermore, the ESS and VB hysteresis temperatures, vacillating between room and oral temperature, are more ideal than P for clinical operating conditions since any deformation that the ESS and VB NiTi-alloys had introduced at room temperature can be easily erased on the application of oral temperature, which is high enough to ensure complete transformation to the austenite phase. However, the austenitic-finish temperature at 47.2^{o}C for P is much greater than oral temperature and therefore is neither clinically conducive nor workable. At room temperature, all three systems (ESS, VB, and P) are equally sensitive to torsional stress since they are mostly in their martensitic phase, which has a lower density and less rigidity than in their austenitic phase.

A criticism of the study design was that it was not conducted at the same temperature as the oral environment. de Vasconcelos et al[16] recommended conducting fatigue tests at the oral temperature. Additionally, the DSC sample sizes were relatively small when compared to similar studies.

Conclusion:

Irrespective of thermal history, files with high TMs conveyed low RDs. Heat-treated files displayed higher cyclic-fatigue resistance than conventional files under similar 100-600 RPM range and have an endurance limit of RPM=100, below which N was theoretically infinite. RPM appeared independent of N for conventional files.

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