



## Comparative Evaluation of Super-elastic behavior and Phase transformation of used and unused Rotary Nickel-Titanium Endodontic Instruments from Different Manufacturers: A Differential Scanning Calorimetric (DSC) study

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### ABSTRACT

Differential Scanning Calorimetric (DSC) study was utilized to investigate the Super-elastic behavior and Phase transformation of used and unused Rotary Nickel-Titanium (NiTi) endodontic instruments from 3 different manufacturers in the as-received condition and after subjection to 3 sterilization cycles. A total of 72 ProTaper (n=24), Mtwo (n=24) and HyFlex (n=24) files were examined. DSC measurements showed that among the specimens of all three brands, in the as-received condition and after 3 sterilizations, Hyflex rotary files has better super elasticity than the ProTaper and Mtwo rotary files. It has advantageous microstructure and a better manufacturing process as the temperature range for phase transformation is lowest for HyflexNiTi rotary files.

**Keywords:** Super-elastic, Rotary Nickel-Titanium, DSC, phase transformation.

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### INTRODUCTION

*"Darkness cannot drive out darkness: only light can do that"-Martin Luther King Jr.*

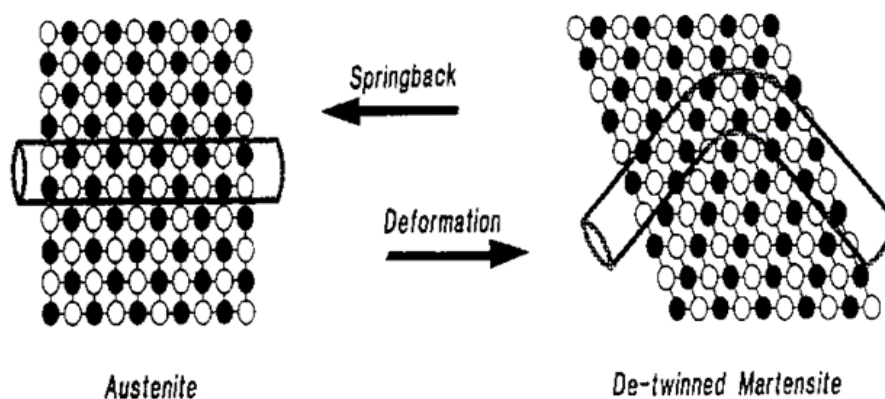
After the pioneering research by Walia H, which introduced nickel-titanium (NiTi) hand files to the endodontic profession, both nickel-titanium hand files and particularly rotary instruments have achieved widespread popularity. A major reason for their selection is the much greater flexibility (i.e. much lower elastic modulus) of the nickel-titanium alloy compared with stainless steel, which offers distinct clinical advantages with curved root canals [1-4].

Nitinol alloys exhibit two closely related and unique properties: shape memory and Super elasticity (also called pseudo elasticity). Nickel-Titanium (NiTi) endodontic files show greater flexibility and superior resistance in bending and torsion compared with stainless steel instruments. Some researchers found that heat sterilization leads to reduction of cutting efficiency of the NiTi files.<sup>12</sup> Using a spectroscope, Shabalovskaya and Anderegg examined NiTi alloy surfaces after repeated sterilization cycles. They noticed that autoclaving at 120°C and 21psi (pound per square inch) resulted in an alteration of alloy surface. In particular, files that underwent five cycles of sterilization experienced a reduction in cutting ability of approximately 16.1% compared with untreated files. After 10 cycles of sterilization, the cutting efficiency was further reduced by up to 50.8% [5-9].

The NiTi alloy's inherent ability to undergo phase transformation within NiTi matrix, from austenite (parent phase) to martensite (daughter phase) with application of temperature or stress, giving rise to Shape Memory Effect (SME) and Superelasticity (SE). Differential Scanning Calorimetry (DSC) has been successfully used to investigate phase transformation within NiTi matrix of popular brands of endodontic instruments [10-11].

The martensite shape can be deformed easily to a single orientation by a process known as detwinning. NiTi alloy is more ductile in the martensitic phase than the austenite phase. The deformation can be reversed by heating the alloy above the TTR i.e. the reverse transformation temperature range or RTTR with the result that the properties of the NiTi alloy revert back to their previous higher temperature values. The alloy resumes the original parent structure and orientation as the body-centred cubic, high temperature phase termed *austenite* with a stable energy condition. The transition from the

austenitic to martensitic phase as a result of the application of stress, such as occurs during root canal preparation, in NiTi alloy a *stress-induced martensitic transformation* occurs [12-16].



**Diagrammatic representation of the super-elasticity effect of NiTi alloy.**

DIFFERENTIAL SCANNING CALORIMETRY or DSC is a thermo-analytical technique in which it measures the temperatures and heat flows associated with phase transitions in materials as a function of time and temperature in a controlled atmosphere. These measurements provide quantitative and qualitative information about physical and chemical changes that involve endothermic or exothermic processes, or changes in heat capacity [17-19].

The basic principle underlying this technique is that, when the sample undergoes a physical transformation such as *phase transitions*, more or less amount of heat will need to flow to it. Whether less or more heat need to flow to the sample depends on whether the process is *exothermic* or *endothermic* [20].

In this study we have discussed about Superelastic quality of nickel-titanium rotary endodontic instruments of different makers utilizing the measurement obtained through different phase relationships of a controlled heat treatment procedure namely Differential Scanning Calorimetry (DSC) experiment. The endodontic instruments used were Dentsply Maillefer- ProTaper rotary file system, Coltene Whaledent- Hyflex CM file system and VDW Selmark-Mtwo file system. The files were used in as received condition i.e. new or unpacked and after three cycle of sterilization (autoclaved at 121° C at 15psi for 15 min). From this we can have an idea about the flexibility of these different files i.e. the file system which will have less DSC value will have better superelastic behavior & will show better flexibility. This will help us to choose the rotary file system we will prefer for the endodontic treatment mainly where the canals are curved [21-13].

## MATERIAL AND METHODS

The study was conducted in Guru Nanak Institute of Dental Science & Research, Kolkata & Institute of Minerals and Materials Technology (IMMT), Bhubaneswar.

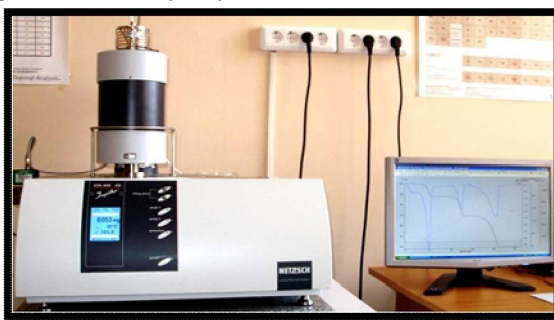
**Table 1. SAMPLE SIZE**

Manufacturer's names:	Types of files	Taper of files	Length of Files	No of files	
				A	B
Dentsply Maillefer- ProTaper file system	Size- 25	0.06	25 mm	6	6
	Size- 35	0.04	25 mm	6	6
Coltene Whaledent- Hyflex CM file system	Size-25	0.04	25 mm	6	6
	Size-40	0.04	25 mm	6	6
VDW Selmark-	Size-25	0.06	25 mm	6	6
Mtwo file system	Size-35	0.04	25 mm	6	6

[A=NEW, B= AFTER 3 CYCLE STERILIZATION]

**STUDY TOOLS:**

1. Differential Scanning Calorimetric (DSC) Machine:



**Netzsch STA 449 F3 Jupiter**

2. Slow-speed diamond saw



**BUEHLER IsoMet Low Speed Saw**

3. Special computer setup attached with the DSC machine
4. Liquid Nitrogen cylinder
5. X-Smart Endodontic micro-motor
6. 2L Class N Tabletop Steam Autoclave
7. 2.5% Sodium hypochlorite
8. Stainless Steel Autoclave Box
9. Distilled water

**STUDY METHODOLOGY:**

**Sample Preparation-**

- Endodontic rotary NiTi files taken as test samples were collected from 3 different manufactures i.e. ProTaper file system, Mtwo file system and Hyflex file system.
- From ProTaper file system size 25 and 35, from Mtwo file system size 25 and 35 and from Hyflex file system size 25 and 40 (Hyflex file system don't have size 35) were taken.
- 12 no of files from each size were taken.
- This means total of 24 no files were taken from each file system, so the total no of files taken for test were 72.
- From the 12 no of files of each size 50% of it i.e. 6 no of files were tested new or as received condition and rests 6 no of files were tested after 3 applications and subsequent sterilization.
- Each application i.e. biomechanical preparation with the help of X-Smart Endodontic micro-motor was done for 3 min in a extracted maxillary central incisor followed by sterilization. This procedure was repeated for another 2 times for the same file, so total 9 min of application and 3no of subsequent sterilization.

**Sample Sterilization-**

- For sterilization each file after 3 min application was first kept in 2.5% Sodium hypochlorite (NaOCl) for 15min.
- Then rinsed thoroughly under distilled water & kept overnight to dry before sterilization procedure.
- Next, files were kept in autoclave box and autoclaved at 121°C at 15 pound per square inch (psi) for 15min in Autoclave followed by overnight drying.

**Sample Testing:**

- For all the file samples Differential Scanning Calorimetric (DSC) experiment was done in Netzsch STA 449 F3 Jupiter machine between 25°C to 75°C.
- The linear heating rate is a standard 5°C per minute.
- The DSC result was monitored in the computer program which was attached to it.
- The results were noted and the DSC value to each sample was compared.
- The results were then sent for statistical analysis.

**RESULTS**

The samples Ni-Ti endodontic rotary files used for this study were collected from different manufacturers and tested in the Differential Scanning Calorimetric machine to study their phase transformation and evaluate their super elastic behavior. The results and observations thus derived are as follows:-

- Table 1 & Table 2 shows the Mean Mass & DSC value of unused & used ProTaper Size 25 with Taper 0.06 NiTi Rotary files.
- In Table 1 the files were as received condition & in Table 2 the files were after 3 applications and subsequent sterilization.
- The mean value of both mass & DSC value is calculated.

**Table 2. ProTaperNiTi Rotary file system (DentsplyMaillefer, Switzerland)  
Group 1: Size 25 Taper 0.06- New or as received condition**

	Mass in mg unit	DSC in m.cal/sec unit
Mean	4.59	0.47 to -3.33

**Table 3. ProTaperNiTi Rotary file system (DentsplyMaillefer, Switzerland)  
Group II: Size 25 Taper 0.06- After 3 cycle of sterilization**

	Mass in mg unit	DSC in m.cal/sec unit
Mean	4.52	0.47 to -3.23

- Table 3 & Table 4 shows the Mean Mass & DSC value of unused & used Protaper Size 35 with Taper 0.04 NiTi Rotary files.
- In Table 3 the files were as received condition & in Table 4 the files were after 3 applications and subsequent sterilization.
- The mean value of both mass & DSC value is calculated.

**Table 4. ProTaperNiTi Rotary file system (DentsplyMaillefer, Switzerland)  
Group III: Size 35 Taper 0.04- New or as received condition**

	Mass in mg unit	DSC in m.cal/sec unit
Mean	4.85	0.50 to -3.28

**Table 5. ProTaperNiTi Rotary file system (DentsplyMaillefer, Switzerland)  
Group IV: Size 35 Taper 0.04- After 3 cycle of sterilization**

	Mass in mg unit	DSC in m.cal/sec unit
Mean	4.73	0.5 to -3.24

- Table 5 & Table 6 shows the Mean Mass & DSC value of unused & used Mtwo Size 25 with Taper 0.06 NiTi Rotary files.
- In Table 5 the files were as received condition & in Table 6 the files were after 3 applications and subsequent sterilization.
- The mean value of both mass & DSC value is calculated.

**Table 6. MtwoNiTi Rotary file system (VDW, Munich, Germany)  
Group V: Size 25 Taper 0.06- New or as received condition**

	Mass in mg unit	DSC in m.cal/sec unit
Mean	4.61	0.47 to -3.33

**Table 7. MtwoNiTi Rotary file system (VDW, Munich, Germany)  
Group VI: Size 25 Taper 0.06- After 3 cycle of sterilization**

	Mass in mg unit	DSC in m.cal/sec unit
Mean	4.61	0.47 to -3.33

- Table 7 & Table 8 shows the Mean Mass & DSC value of unused & used Mtwo Size 35 with Taper 0.04 NiTi Rotary files.
- In Table 7 the files were as received condition & in Table 8 the files were after 3 applications and subsequent sterilization.
- The mean value of both mass & DSC value is calculated.

**Table 8. MtwoNiTi Rotary file system (VDW,Munich,Germany)  
Group VII: Size 35 Taper 0.04- New or as received condition**

	Mass in mg unit	DSC in m.cal/sec unit
<b>Mean</b>	<b>4.59</b>	<b>0.47 to -3.4</b>

**Table 9. MtwoNiTi Rotary file system (VDW,Munich,Germany)  
Group VIII: Size 35 Taper 0.04- After 3 cycles of sterilization**

	Mass in mg unit	DSC in m.cal/sec unit
<b>Mean</b>	<b>4.63</b>	<b>0.45 to -3.11</b>

- Table 9 & Table 10 shows the Mean Mass & DSC value of unused & used Hyflex CM Size 25 with Taper 0.04 NiTi Rotary files.
- In Table 9 the files were as received condition & in Table 10 the files were after 3 applications and subsequent sterilization.
- The mean value of both mass & DSC value is calculated.

**Table 10. HYFLEX® CM NiTi Rotary file system (Coltene/Whaledent,Ohio,USA)  
Group IX: Size 25 Taper 0.04- New or as received condition**

	Mass in mg unit	DSC in m.cal/sec unit
<b>Mean</b>	<b>4.85</b>	<b>0.54 to -3.33</b>

**Table 11. HYFLEX® CM NiTi Rotary file system (Coltene/Whaledent,Ohio,USA)  
Group X: Size 25 Taper 0.04- After 3 cycle of sterilization**

	Mass in mg unit	DSC in m.cal/sec unit
<b>Mean</b>	<b>4.73</b>	<b>0.48 to -3.29</b>

- Table 11 & Table 12 shows the Mean Mass & DSC value of unused & used Hyflex CM Size 40 with Taper 0.04 NiTi Rotary files.
- In Table 11 the files were as received condition & in Table 12 the files were after 3 applications and subsequent sterilization.
- The mean value of both mass & DSC value is calculated.

**Table 12. HYFLEX® CM NiTi Rotary file system (Coltene/Whaledent,Ohio,USA)  
Group XI: Size 40 Taper 0.04- New or as received condition**

	Mass in mg unit	DSC in m.cal/sec unit
<b>Mean</b>	<b>4.59</b>	<b>0.51 to -3.31</b>

**Table 13. HYFLEX® CM NiTi Rotary file system (Coltene/Whaledent,Ohio,USA)  
Group XII: Size 40 Taper 0.04- After 3 cycles of sterilization**

	Mass in mg unit	DSC in m.cal/sec unit
<b>Mean</b>	<b>4.52</b>	<b>0.5 to -3.2</b>

Statistical analysis for the different groups are presented by Mean, Standard deviation (s.d.), Degree of Freedom (d.f.), Test of significance (t), Probability (p) & More than (>). In order to compare the all 3 different rotary file system's DSC experiment value a one-way analysis of variance i.e. the variance (F) ratio test was conducted. The test was considered significant as the F-value obtained differ significantly at 5% level(Mtwo differs significantly from Hyflex&Protaper).

First the mean±S.D (mass value) of new instrument compared with instrument after 3-cycle of use. For the purpose of test of significance, paired t- test used and the result has been assessed through the P-value.

Test of significance shows that the difference in between new and after 3 cycles of use of the instruments do not differ significantly with p value>0.05.

The t-value was calculated applying the formula  $t = \bar{D}/S.E.$  where  $S.E. = S.D./n^{1/2}$

$\bar{D}$ : average of deviation (new & after  
 3 cycle of sterilization Tabulated t value, 5df, 5% level =2.57)  
 S.E.: Standard error  
 S.D: Standard deviation of the  
 differences.

**Tabulated t value, 5df, 5% level =2.57**

Then the mean  $\pm$ S.D (DSC value) of new instrument has been compared with instrument after 3-cycle of use. For the purpose of test of significance, paired t- test used and the result has been assessed through the P-value. Test of significance shows that the difference in between new and after 3 cycles of use of the instruments pro-taper size -25, size-35, Mtwo size 25 & 35, Hyflex size 25 and size 40 do not differ significantly with p value > 0.05. However Mtwo size 35 differ significantly with p value < 0.05.

**Tabulated t value, 5df, 5% level =2.57**

The difference  $\pm$  SD (DSC) between new and after 3 cycle of different instrument has been compared. While comparing Protaper with Mtwo test of significance shows that Protaper differs significantly from Mtwo with p value less than 0.05 but with Hyflex, test of significance shows that both the instruments do not differ significantly with p value more than 0.05. Comparing Mtwo with Hyflex test of significance shows that both the instruments differ significantly with p value less than 0.05 with respect to the super elastic behavior and phase transformation.

**Tabulated t value 22d.f, 5% level = 2.07**

Difference in between new and after 3 cycles DSC of different instruments, viz- Mtwo, Hyflex and Protaper has been calculated. Mean differences of each instruments and their square deviations has been calculated to find out the total sum of square, sum of square between the classes and sum of square between the classes for the purpose of Analysis of variance.

$D_i = \text{Difference in between new \& after 3-cycle of sterilization}$   
 $\sum D_i = \sum D_1 + \sum D_2 + \sum D_3 = 2.841 + 0.469 + 0.903 = 4.213$   
 Total sum of square = 1.039

Name	mean diff
Pro Taper	0.075
M Two	0.236
Hyflex	0.039

Sum of square between the classes = 0.264  
 Error sum of square = 0.775

**Analysis of variance (F) ratio test**

Square of Variances	d.f	Sum of Square	Mean sum of Square	F-Ratio
Between classes	3-1=2	0.264	0.132	5.73
Error	35-2=33	0.775	0.023	
Total	36-1=35	1.039		

➤ Compound 'F' Ratio =  $\frac{0.132}{0.023} = 5.73$

Table F- Value at 2df ( → ) and 33df ( ) at 5% level of significance is 3.23. Since the compound F-ratio is more than the tabulated F-ratio, the difference of DSC (New & after 3- cycle), between the instruments of different manufactures differ significantly at 5% level.

**Mean DSC value for the 3 different rotary endodontic NiTi files systems are**

From the mean DSC values significant differences are revealed between files of Mtwo rotary files and Protaper and Hyflex rotary files. Hyflex rotary files showed less mean difference than the other two file systems which signifies that the Hyflex file system has a advantageous microstructure with better superelastic behavior. For Hyflex the transformation from martensitic to austenitic NiTi is completed between room temperature and oral environment temperature (~37°C) while for Protaper and Mtwo the transformation temperature value range is higher. The mean difference value between Hyflex and Protaper is less comparative to the mean value for Mtwo which is much higher than both the Hyflex and Protaper [22-29].

## DISCUSSION

The file systems had gone under various tests by different scientists to know about their superelasticity, shape memory, change in microstructure, effect of sterilization, etc but thermal analysis for these file systems reference not available. Among different thermal analysis tests like differential thermal analysis (DTA), thermogravimetry (TGA), evolved gas analysis (EGA) and differential scanning calorimetry (DSC) the DSC is a powerful tool for materials characterization of rotary instruments, providing direct information not readily available from other analytical techniques about the NiTi phases present, which are fundamentally responsible for their clinical behavior. Studies by *W. A. Brantley et al.*,<sup>22</sup>Svec TA, Iijima M, et al [20], Georgia B. Alexandrou et al,[32] also supports this theory that DSC is ideal test to determine the physiological & mechanical properties of the files.

In this present study which is more importantly is an attempt to evaluate the superelastic behavior of the NiTi rotary endodontic files by DSC test of three different manufacturers namely ProTaperNiTi Rotary file system (DentsplyMaillefer, Switzerland), MtwoNiTi Rotary file system (VDW, Munich, Germany) and HYFLEX® CMNiTi Rotary file system (Coltene/Whaledent, Ohio, USA) in relation to the phase transformation. Also in combine this study will be first of its kind to test the super elasticity of these three different file systems.

The result of our study can be cross matched with the results obtained by *T. A. Svec et al.* [31], where they have evaluated flexibility of two rotary instruments of different manufacturers by DSC method.

According to *W. A. Brantley et al.*, in their study [22] based on the DSC value they told that Light speed instruments have a better flexibility with advantageous microstructure than Profile instruments [29-32]. The use of sodium hypochlorite (NaOCl) to irrigate root canals is currently the gold standard to achieve tissue dissolution and disinfection. From the study they concluded that it would be a risk of corrosion of NiTi rotary files in contact with NaOCl. Under the conditions of this in vitro study, both ProFile and RaCe rotaries showed a reduction in fatigue resistance but not in torsional strength. Analysis & critical discussion of the facts and figures derived from the study helps to clearly resolve that Hyflex file system has better superelasticity than the ProTaper and Mtwo file system [32-36]. It has advantageous microstructure and a better manufacturing process as the temperature range for phase transformation is lowest for HyflexNiTi rotary files. As in our study, a limited variable was taken and only two sizes of rotary files were included to conduct the DSC, further studies might enlighten the greater knowledge and understanding about the subject studied and signify & support the result of our study.

## CONCLUSIONS

The results of this in-vitro study guided to draw the following conclusions:

- The DSC thermal analysis is a powerful tool to examine the superelastic behavior of the NiTi rotary endodontic files.
- The NiTi rotary endodontic files from Coltene i.e. Hyflex CM has better superelastic behavior than Protaper and MtwoNiTi rotary files.
- By analyzing statistically, using the variance 'F' test, it was found that the mean DSC value for ProTaper was 0.075, for Mtwo was 0.236 & for Hyflex was 0.039.
- The result that the microstructure of Mtwo files are significantly different than Protaper and Hyflex.
- While comparing ProTaper with Hyflex, test of significance shows that both the instruments do not differ significantly in terms of their flexibility.

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