



Fatigue resistance of ProTaper gold exposed to high-concentration sodium hypochlorite in double curvature artificial canal

Fahda Algahtani^{a,b,1}, Xiangya Huang^{c,a,1}, Markus Haapasalo^a, Zhejun Wang^a, Ahmed Hieawy^a, Duo Zhang^a, Jolanta Aleksejuniene^a, Ya Shen^{a,*}

^a Department of Oral Biological and Medical Sciences, Faculty of Dentistry, The University of British Columbia, Vancouver, Canada

^b College of Dentistry, Princess Nourah Bint Abdulrahman University, Saudi Arabia

^c Department of Conservative Dentistry and Endodontics, Guanghua School of Stomatology, Sun Yat-Sen University, Guangzhou, China

ARTICLE INFO

Keywords:

Body temperature
Corrosion
Double curvature
Fatigue resistance
ProTaper Gold

ABSTRACT

This study aimed to evaluate and compare the fatigue resistance of ProTaper Gold (PTG) and ProTaper Universal (PTU) in artificial single and double curvature canals in 5% sodium hypochlorite (NaOCl) at body temperature (37 °C). PTG and PTU files (size F1) were subjected to fatigue tests in two different artificial ceramic canals. The single curvature model had a 60° curvature angle with a 5 mm radius. The double curvature model had a 60° curvature angle with a 5 mm radius and a second 30° curvature with a 2 mm radius. A file segment was introduced into the artificial canal and immersed in water or 5% NaOCl at 37 °C. The total number of cycles to fracture (NCF) was recorded. Data were analyzed using *t*-test and linear regression analysis. The NCF of all files was significantly influenced by the type of NiTi metal alloy ($P < .01$), canal curvatures ($P < .01$), and the environmental conditions ($P < .05$). PTG had higher fatigue resistance than PTU files in both single and double curvature canals ($P < .05$). The NCF of PTU files in 5% NaOCl was shorter than that in water ($P < .05$). The mean length of broken PTG was significantly shorter than those of PTU files in both single and double curvature canals ($P < .01$). The fatigue performance of PTG is better than that of PTU in both single and double curvature. Environmental conditions may affect the fatigue behavior of PTU files with single curvature.

1. Introduction

S-shaped or bayonet shaped canals pose great problems during endodontic therapy, since they involve at least two curves, with the apical curve having maximum deviations in anatomy [1,2]. Endodontic cleaning and instrumentation is often regarded as technically demanding and difficult when such systems are presented. Those systems pose high stress and dangerous anatomy for nickel-titanium (NiTi) rotary instruments with an increased risk of instrument fracture. Two different mechanisms may lead to NiTi rotary fracture: cyclic fatigue and torsional fracture. When a rotary file undergoes repeated compression and extension in a curved canal, this can cause work hardening of the metal, which causes cyclic fatigue and an increased risk of fracture [3]. Fatigue has been implicated as the main reason for the fracture of endodontic rotary files in clinical use [4,5].

ProTaper Universal (PTU, Dentsply Tulsa Dental Specialities, Tulsa,

OK) is a much studied NiTi rotary system manufactured with a variable taper over the length of the cutting blades, convex triangular cross sections, and noncutting tips. Later, ProTaper Gold (PTG, Dentsply Tulsa Dental Specialities) instruments were introduced. The PTG files have geometries identical to PTU but are more flexible and have been developed with proprietary advanced metallurgy. Heat treatment (thermal processing) is one of the most fundamental approaches toward adjusting the transition temperatures of NiTi alloys [6–9] and affecting the fatigue resistance of NiTi endodontic files. Due to their specific phase transformation behavior the PTG instruments have a higher austenite finish temperature than the PTU instruments. Recent studies [9–11] showed that the fatigue resistance of PTG was superior to PTU in single curvature canal. So far there is no information available on fatigue resistance of double curvature canal on PTG instruments.

Root canal instrumentation is recommended to be performed with sodium hypochlorite (NaOCl) as an irrigant in the canal(s) and a

Peer review under responsibility of KeAi Communications Co., Ltd.

* Corresponding author. Division of Endodontics Department of Oral Biological & Medical Sciences, UBC Faculty of Dentistry, 2199 Wesbrook Mall, Vancouver, BC, V6T 1Z3, Canada.

E-mail address: yashen@dentistry.ubc.ca (Y. Shen).

¹ F. Algahtani and X. Huang contributed equally to this work.

<https://doi.org/10.1016/j.bioactmat.2019.07.003>

Received 26 March 2019; Received in revised form 10 July 2019; Accepted 30 July 2019

Available online 09 September 2019

2452-199X/ This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

reservoir in the pulp chamber. NaOCl has antimicrobial and tissue-dissolving activity [12,13], but can also cause the corrosion of metals. Corrosion resistance of NiTi instruments is controversial [14–21]. However, most previous studies were performed after passive exposure of the instruments to NaOCl for different time periods [14,15,18,19] immediately before the fatigue test or while instruments were immersed in a low concentration of NaOCl (ie, 1.2%) [16,17] to avoid the corrosion of the test models, which typically are or contain metallic parts such as stainless steel. Currently, a couple of studies [21–24] have shown that immersion in water or NaOCl at simulated body temperature was associated with a marked decrease in the fatigue life of heat treated NiTi files, compared to the room temperature. Therefore, fatigue resistance should be tested under specific temperature conditions. The purpose of this study was to evaluate the corrosion effect of 5% NaOCl on PTG and PTU instruments at body temperature and to examine the fatigue behavior of NiTi files in the custom-made zirconium oxide model with single and double curvature canals.

2. Materials and methods

Instruments of PTU and PTG (F1) were subjected to fatigue tests inside two custom-made zirconium oxide canals. Each group included 15 instruments. The artificial ceramic canals were milled in an InCoris ZI zirconium oxide disc (Dentsply Sirona, Bensheim, Germany) using the inLab MC X5 Digital computeraided design and computer-aided manufacturing (CAD/CAM) System (Dentsply Sirona). The canal was customized to apical size #25, and apical tapers from 0 to 6 mm was 8%, middle tapers from 6 to 13 mm was 6% and coronal tapers from 13 to 16 mm was zero taper. The single curvature model had a 60° curvature angle and 5 mm radius. In the double curvature model, the first angle was a 60° curvature angle with a 5 mm radius and the second angle was a 30° curvature with a 2 mm radius [25]. The model was fixed and placed in 300 mL of 5% NaOCl (The Clorox Company, Brampton, Ontario, Canada) or distilled water. The container was mounted on a hot plate using plastic strips, and the temperature of the medium solution was stabilized to remain at body temperature (37 °C). A 19-mm-long segment from the tip of the instrument was introduced into the ceramic canal and immersed in the liquid medium during the test [20]. The torque control motor (AEU-20T Endodontic System) settings for the rotary handpiece were adjusted to follow the manufacturer recommendation for size F1 ProTaper files (PG: 300 rpm and 150 GCM, PU: 300 rpm and 250 GCM). The time to fracture (seconds) was recorded and multiplied by the number of rotations per minute to obtain the total number of cycles to failure (NCF). The length of the fragments was measured by using a stereomicroscope at X10 magnification (Microdissection; Zeiss, Bernried, Germany). Three samples from each group were randomly chosen for fractographic examination under a scanning electron microscope (SEM; Helios NanoLab 650; FEI, Eindhoven, Netherlands) [20].

The IBM SPSS for Windows 25.0 software was used for statistical analyses (IBM, Chicago, IL) and the threshold for significance was set at $P < .05$. The normality distribution and the assumption for the homogeneity of variance were examined using the Kolmogorov-Smirnov test and Levene's test, respectively. The independent sample *t*-test was used to compare the study groups. The multiple linear regression analysis was used to examine the potential predictors/explanatory factors associated with the outcome variable (NCF).

3. Results

Table 1 shows the comparison of the performance of two types of files (PTG and PTU) in two types of curvatures. Two types of comparisons are presented in this table. Vertical comparisons compare two files separately for each of the two mediums and each of the two curvatures. The horizontal comparisons compare the performance of a specific file for each curvature separately between the two mediums. The

performance of PTG (vertical comparisons) was significantly better than PTU in both the single curvature model and in the double curvature model and in two different medium solutions, 5% NaOCl and distilled water ($P < .001$). In the single curvature canal, the PTU files were more resistant to fatigue failure in the distilled water than in 5% NaOCl solution ($P < .03$) (Table 1). There were no significant differences of the fatigue resistance of the PTG files between the two mediums either in the single or in the double curved canals.

Tolerance values for the predictors (file type, curvature, and solution) of the multiple regression model were 1.0, indicating the assumption of absence multicollinearity was fulfilled (Table 2).

The linear regression in this model contains independent variables that are statistically significantly different with a reasonably high R-squared value ($P < .001$), and three predictors jointly explained 75.4% of the variance in the NCF. This analysis showed that the NCF was significantly influenced by file type ($P < .001$), canal curvature ($P < .01$), and the type of medium solution ($P < .05$).

There were significant differences in file lengths between the two types of files (PTG vs. PTU) in both single ($P < .001$) and double curvature ($P < .001$) canals. The fragment of PTG was significantly shorter than those of PTU files in both single curvature (3.6 ± 0.2 mm vs. 5.4 ± 0.3 mm) and double curvature (3.6 ± 0.3 mm vs. 4.4 ± 0.9 mm). The mean length of broken fragments of PTU in single curvature canals (5.4 ± 0.3 mm) was significantly longer than that of fragments in double curvature canal (4.4 ± 0.9 mm) ($P < .001$). The SEM topographic appearance of the fracture surfaces showed typical features of cyclic fatigue (Fig. 1). None of the tested files showed pitting or crevice corrosion in water or 5% NaOCl as evaluated under SEM.

4. Discussion

The size of ProTaper F1 is 20/.07 at the file tip area, while from D4 to D14 the F1 file has a gradually decreasing taper which serves to improve the flexibility of the file. In this study, the custom-made, size-matched artificial canal allowed the instrument to have a close fit without binding. Only one instrument size of both brands (size F1) was tested because this is the most commonly used size during instrumentation. The noncorrosive biomaterial model allowed a standardized evaluation of the fatigue resistance of NiTi files in a potentially corrosive environment of concentrated NaOCl, which will mimic clinical conditions better than earlier models. Recently, only one study [23] evaluated the fatigue resistance of PTG F2 immersed in 2.5% and 5.25% NaOCl using stainless steel block model in a single curvature. The authors described that for the corrosive action of NaOCl, the artificial metallic canal was replaced when any sign of corrosion was observed. To our knowledge, the present study is the first one to simulate the fatigue resistance of PTG files in the continuous presence of a high concentration of NaOCl in the double curvature canal.

Recently, the temperature has been investigated as a possible variable influencing the fatigue resistance of rotary NiTi files [11,21–24]. Thermally treated NiTi files have transformation temperatures much higher than those of conventional austenitic NiTi files [7,9,26]. de Hemptinne et al. [27] found that intracanal temperature ranges from 31 to 35 °C. Some studies [27,28] have shown that warming NaOCl to 60 °C significantly increases the rate and effectiveness of tissue dissolution. It has been reported that the benefits of heated irrigants only lasts during delivery, since body temperature is reached immediately after [29]. In the other way, an irrigant at room temperature is quickly warmed to body temperature when entering the root canal [29]. Therefore, to simulate the clinical situation, the testing temperature was set to 37 °C in the present study.

The metallurgical characteristics of PTU files showed that file has an austenite structure, whereas PTG file with thermal processing is essentially in the martensite condition at body temperature [9]. This would explain why the fatigue resistance of PTG was better than that of PTU files in both single and double curvatures. Interestingly, there was

Table 1

The number of revolutions until fracture of files in water and sodium hypochlorite (NaOCl) at 37 °C with a single curve and double curves.

	Single curvature			Double curvature		
	Water	5% NaOCl	Significance	Water	5% NaOCl	Significance
	Mean ± SD	Mean ± SD		Mean ± SD	Mean ± SD	
ProTaper Gold	667 ± 118	662 ± 118	0.922	591 ± 87	533 ± 73	0.079
ProTaper Universal	343 ± 53	266 ± 106	0.030	303 ± 47	298 ± 62	0.818
Significance	< 0.001	< 0.001		< 0.001	< 0.001	

Table 2

Predictors of file failure (linear multiple regression).

Linear regression model		
Model summary: Adj R ² = 0.754 (p < 0.001)		
Predictors	β(p)	Tolerance
File type	−0.854 (< 0.001)	1.0
Curvature	−0.147 (0.003)	1.0
Solution	−0.100 (0.044)	1.0

no significant difference of fatigue resistance of PTG files between water and 5% NaOCl in both curvatures. Post heat treatment was applied after the flutes of PTG file have been manufactured. The temperature used in heat treatment is in a range of 370–510 °C for a variable period of time (typically 10–60 min, depending on file size and taper) [30]. With increased exposure time at moderate temperatures, the oxide formed is composed mainly of TiO₂ with a slow formation and growth [31]. The observation in the present study further supports the assumption that the oxide surface layer which can be obtained after the

proprietary heat treatment manufacturing process may protect the stressed PTG instruments from corrosion.

The fractographic appearance of instruments fatigued in hypochlorite is very similar to that the instrument tested in deionized water. Intergranular crack growth, typical of corrosion fatigue [32] was not detected here. However, 5% NaOCl had a negative effect on the fatigue life of PTU in the single curvature. The present study focused on the low cyclic fatigue test, which was less than 350 cycles for PTU, and thus the corrosion on the fractographic surface at low strain amplitudes could not be determined. Further research is needed to evaluate the corrosion behavior of NiTi files by the cyclic potentiodynamic polarization curves.

A double curvature canal was simulated in the present study in which the second curvature was extended from the single curvature model. No significant difference of the fatigue life between water of NaOCl on double curvature, which may be partly explained by the short fatigue life on the double curvature, compared to the single curvature. The majority of files in the double curvature fractured in the second curvature area. The explanation for this is that the second curvature was more abrupt than the first curvature due to the short radius of only

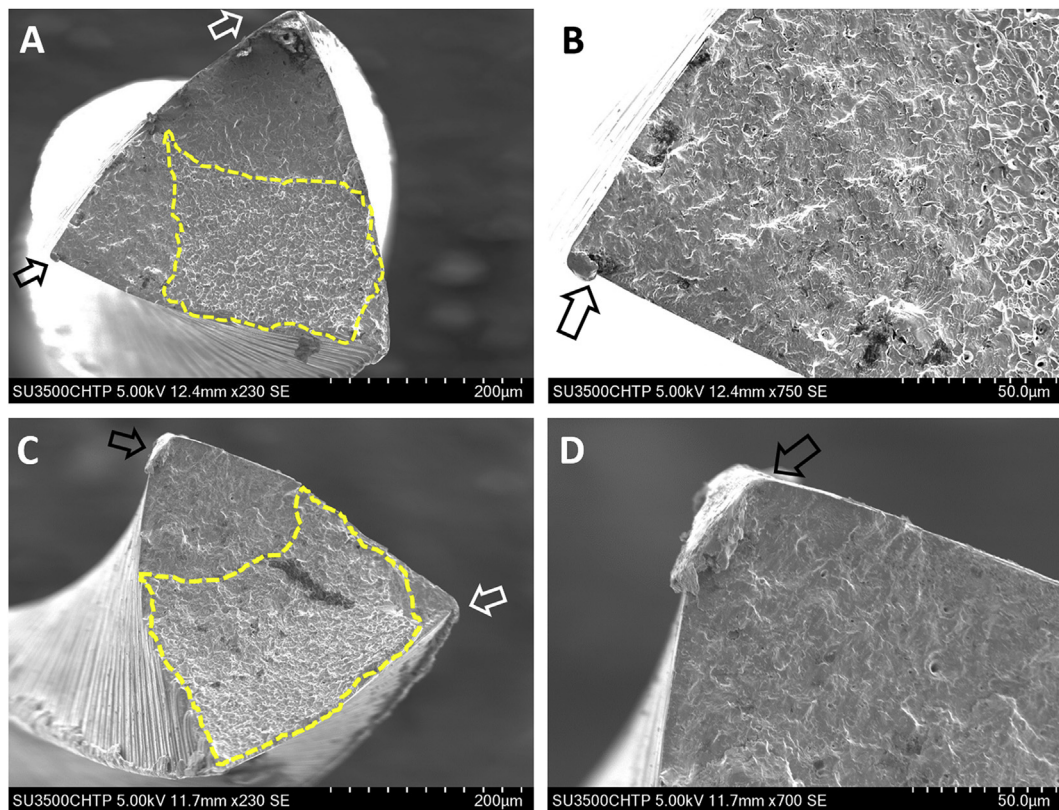


Fig. 1. The fracture surface of ProTaper Gold files after the fatigue test in 5% NaOCl at 37 °C in single curvature (A and B) and double curvature (C and D) canals. High-magnification view of one cutting edge area of the crack ignition in B (black arrow in A) and D (black arrow in C). The arrows point toward the crack initiation points; the region of fatigue crack propagation and dimple area outlined by the dotted line. No signs of corrosion can be seen on the files.

2 mm, which is in agreement with the results obtained in a previous double curvature study [25]. Interestingly, 10 of 30 PTU and 5 of 30 PTG files fractured in the first curvature. This may be caused by the fact that PTU files are stiffer and, consequently, the first and second curvature may have strains under this condition. Previous study [33] showed that the length of fragment tended to affect the success rate of the removal of fractured NiTi instruments. The short fragment may raise the challenge for the removing instruments.

In summary, the two precisely shaped, variable taper artificial curvature canals (one single curvature and one double curvature canals) enabled the characterization of fatigue resistance of PTU and PTG files in high concentration NaOCl. The fatigue performance of PTG is better than that of PTU in both single and double curvature. Double curvature canals represent much more stressful and challenging anatomy than single curvature canals for the variable taper files. The effect of NaOCl on PTU files in use cannot be dismissed.

Acknowledgments

This work was partly supported by Canada Foundation for Innovation (CFI fund # 32623). The authors thank Dentsply Tulsa for donating the files used in this study. The authors deny any conflicts of interest.

References

- [1] C.J. Cunningham, E.S. Senia, A three-dimensional study of canal curvatures in the mesial roots of mandibular molars, *J. Endod.* 18 (1992) 294–300.
- [2] N. Kartal, The degrees and configurations of mesial canal curvatures of mandibular first molars, *J. Endod.* 23 (1997) 358–362.
- [3] J.P. Pruett, D.J. Clement, D.L. Carnes Jr., Cyclic fatigue testing of nickel–titanium endodontic instruments, *J. Endod.* 23 (1997) 77–85.
- [4] G.S.P. Cheung, B. Peng, Z. Bian, Y. Shen, B.W. Darvell, Defects in ProTaper S1 instruments after clinical use: fractographic examination, *Int. Endod. J.* 38 (2005) 802–809.
- [5] Y. Shen, G.S. Cheung, Z. Bian, B. Peng, Comparison of defects in ProFile and ProTaper systems after clinical use, *J. Endod.* 32 (2006) 61–65.
- [6] C. Frick, A. Ortega, J. Tyber, et al., Thermal processing of polycrystalline NiTi shape memory alloys, *Mater. Sci. Eng. A* 405 (2005) 34–49.
- [7] Y. Shen, H. Zhou, L. Campbell, et al., Fatigue and nanomechanical properties of K3XF nickel–titanium instruments, *Int. Endod. J.* 47 (2014) 1160–1167.
- [8] J.L. Gutmann, Y. Gao, Alteration in the inherent metallic and surface properties of nickel–titanium root canal instruments to enhance performance, durability and safety: a focused review, *Int. Endod. J.* 45 (2012) 113–128.
- [9] Y. Shen, H.M. Zhou, Y.F. Zheng, B. Peng, M. Haapasalo, Current challenges and concepts of the thermomechanical treatment of nickel–titanium instruments, *J. Endod.* 39 (2013) 163–172.
- [10] A. Hieawy, M. Haapasalo, H. Zhou, Z.J. Wang, Y. Shen, Phase transformation behavior and resistance to bending and cyclic fatigue of ProTaper Gold and ProTaper Universal instruments, *J. Endod.* 41 (2015) 1134–1138.
- [11] A.D. Uygun, E. Kol, M.K. Topcu, F. Seckin, I. Ersoy, M. Tanriver, Variations in cyclic fatigue resistance among ProTaper Gold, ProTaper Next and ProTaper Universal instruments at different levels, *Int. Endod. J.* 49 (2016) 494–499.
- [12] G. Plotino, N.M. Grande, M. Mercadé Bellido, L. Testarelli, G. Gambarini, Influence of temperature on cyclic fatigue resistance of ProTaper Gold and ProTaper Universal rotary files, *J. Endod.* 43 (2017) 200–202.
- [13] R.E. Hand, M.L. Smith, J.W. Harrison, Analysis of the effect of dilution on the necrotic tissue dissolution property of sodium hypochlorite, *J. Endod.* 4 (1978) 60–64.
- [14] M. Haapasalo, Z. Wang, Y. Shen, A. Curtis, P. Patel, M. Khakpour, Tissue dissolution by a novel multisonic ultracleaning system and sodium hypochlorite, *J. Endod.* 40 (2014) 1178–1181.
- [15] E. Berutti, E. Angelini, M. Rigolone, G. Migliaretti, D. Pasqualini, Influence of sodium hypochlorite on fracture properties and corrosion of ProTaper Rotary instruments, *Int. Endod. J.* 39 (2006) 693–699.
- [16] O.A. Peters, J.O. Roehlike, M.A. Baumann, Effect of immersion in sodium hypochlorite on torque and fatigue resistance of nickel–titanium instruments, *J. Endod.* 33 (2007) 589–593.
- [17] G.S. Cheung, Y. Shen, B.W. Darvell, Does electropolishing improve the low-cycle fatigue behavior of a nickel–titanium rotary instrument in hypochlorite? *J. Endod.* 33 (2007) 1217–1221.
- [18] G.S. Cheung, B.W. Darvell, Low-cycle fatigue of rotary NiTi endodontic instruments in hypochlorite solution, *Dent. Mater.* 24 (2008) 753–759.
- [19] E. Pedullà, N.M. Grande, G. Plotino, A. Pappalardo, E. Rapisarda, Cyclic fatigue resistance of three different nickel–titanium instruments after immersion in sodium hypochlorite, *J. Endod.* 37 (2011) 1139–1142.
- [20] Y. Shen, W. Qian, H. Abtin, Y. Gao, M. Haapasalo, Effect of environment on fatigue failure of controlled memory wire nickel–titanium rotary instruments, *J. Endod.* 38 (2012) 376–380.
- [21] X. Huang, Y. Shen, X. Wei, M. Haapasalo, Fatigue resistance of nickel–titanium instruments exposed to high concentration hypochlorite, *J. Endod.* 43 (2017) 1847–1851.
- [22] R.A. de Vasconcelos, S. Murphy, C.A. Carvalho, R.G. Govindjee, S. Govindjee, O.A. Peters, Evidence for reduced fatigue resistance of contemporary rotary instruments exposed to body temperature, *J. Endod.* 42 (2016) 782–787.
- [23] H. Alfawaz, A. Alqedairi, H. Alsharekh, E. Almuzaini, S. Alzahrani, A. Jamlah, Effects of sodium hypochlorite concentration and temperature on the cyclic fatigue resistance of heat-treated nickel–titanium rotary instruments, *J. Endod.* 44 (2018) 1563–1566.
- [24] Y. Shen, X. Huang, Z. Wang, X. Wei, M. Haapasalo, Low environmental temperature influences the fatigue resistance of nickel–titanium files, *J. Endod.* 44 (2018) 626–629.
- [25] F. Duke, Y. Shen, H. Zhou, et al., Cyclic fatigue of ProFile Vortex and Vortex Blue nickel–titanium files in single and double curvatures, *J. Endod.* 41 (2015) 1686–1690.
- [26] Y. Shen, H.M. Zhou, Y.F. Zheng, L. Campbell, B. Peng, M. Haapasalo, Metallurgical characterization of controlled memory wire nickel–titanium rotary instruments, *J. Endod.* 37 (2011) 1566–1571.
- [27] F. de Hemptinne, G. Slaus, M. Vandendael, W. Jacquet, R.J. De Moor, P. Bottenberg, In vivo intracanal temperature evolution during endodontic treatment after the injection of room temperature or preheated sodium hypochlorite, *J. Endod.* 41 (2015) 1112–1115.
- [28] E. Berutti, R. Marini, A scanning electron microscopic evaluation of the debridement capability of sodium hypochlorite at different temperatures, *J. Endod.* 22 (1996) 467–470.
- [29] G. Sirtes, T. Waltimo, M. Schaetzle, et al., The effects of temperature on sodium hypochlorite short-term stability, pulp dissolution capacity, and antimicrobial efficacy, *J. Endod.* 31 (2005) 669–671.
- [30] D. Sonntag, W.H. Raab, E. Martin, R. Keppel, Intracanal use of heated rinsing solutions: a pilot study, *Quintessence Int.* 48 (2017) 281–285.
- [31] Gao Y, Maxwell R. Endodontic Rotary Instruments Made of Shape Memory Alloys in Their Martensitic State and Manufacturing Methods. United States Patent Application 20110271529 A1.
- [32] D. Hull, *Fractography: Observing, Measuring and Interpreting Fracture Surface Topography*, Cambridge University Press, Cambridge, UK, 1999.
- [33] Y. Shen, B. Peng, G.S. Cheung, Factors associated with the removal of fractured NiTi instruments from root canal systems, *Oral Surg. Oral Med. Oral Pathol. Oral Radiol. Endod.* 98 (2004) 605–610.