Multiple Autoclave Cycles Affect the Surface of Rotary Nickel-Titanium Files: An Atomic Force Microscopy Study

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Abstract

The purpose of this study was to evaluate the surface of rotary nickel-titanium (Ni-Ti) files after multiple autoclave cycles. Two different types of rotary Ni-Ti (Greater Taper and ProFile) were attached to a glass base. After 1, 5, and 10 autoclave cycles the files were positioned in the atomic force microscope. The analyses were performed on 15 different points. The same files were used as control before any autoclave cycle. The following vertical topographic parameters were measured: arithmetic mean roughness, maximum height, and root mean square. The differences were tested by analysis of variance with Tukey test. All topographic parameters were higher for both Greater Taper and ProFile after 10 cycles compared with the control ($P < .05$). ProFile also showed higher topographic parameters after 5 cycles compared with the control ($P < .05$). The results indicated that multiple autoclave cycles increase the depth of surface irregularities located on rotary Ni-Ti files. *(J Endod 2008;34:859–862)*

Key Words

Atomic force microscopy, autoclave, endodontic instruments, rotary nickel-titanium files, sterilization

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Rotary nickel-titanium (Ni-Ti) files are useful instruments for root canal preparation because of their good clinical and mechanical properties (1). Because of high cost, rotary Ni-Ti files are frequently reused after autoclave sterilization. Autoclaving is important to minimize the risk of cross-infection during endodontic treatment, which has been discussed in the literature (2, 3). Despite this fact, no general agreement exists regarding the effect of repeated autoclave cycles on surface of rotary Ni-Ti files (4–7). Surface corrosion could influence mechanical properties of rotary Ni-Ti files and lead to undesirable fracture during root canal instrumentation (8–10).

The surface of a wide variety of materials, including commercial rotary Ni-Ti files, has been characterized by using atomic force microscope (AFM) (11–15). AFM is part of the scanning probe microscopy family that is able to reconstruct, in real time, 3-dimensional surfaces of samples on a computer screen. Moreover, AFM records the data of samples in digital form as sets of x, y, and z values. These sets can be analyzed with dedicated digital software to give all the data pertaining to the examined surface in quantitative form by using vertical topographic parameters (11, 13). Parameters commonly used for describing the topographic characteristics of a surface are the arithmetic mean roughness (AMR), maximum height (MH), and root mean square (RMS) (12, 14, 15). With these parameters it is possible to evaluate vertical amplitude of a surface topography. Therefore, AFM should be ideally suited to the study of autoclave effect on surface topography of rotary Ni-Ti files.

The aim of this study was to investigate the effect of multiple autoclave cycles on vertical amplitude of the surface topography of commercial rotary Ni-Ti files evaluated by using AFM.

Materials and Methods

The analyses were performed on the following commercial rotary Ni-Ti files: Greater Taper (0.06/20, lot 060999740; Dentsply Tulsa Dental, Tulsa, OK) and ProFile (0.06/20, lot 131738; Dentsply Maillefer, Ballaigues, Switzerland).

The active sections of rotary Ni-Ti files were attached to a glass base with rapid-setting cyanoacrylate glue and exposed to multiple autoclave cycles. Each cycle was performed at 121°C, with a pressure of 15 psi, for 15 minutes. The instruments were allowed to dry and cool at room temperature. After 1, 5, and 10 cycles the file samples were positioned in the AFM. The analyses were performed on 15 different points located along a 6-mm section starting at the tip of the file sample. The same rotary Ni-Ti files were used as control before any autoclave cycle (0 cycles).

AFM images of file samples were recorded in contact operation mode on a Shimadzu SPM-9600 AFM (Shimadzu Co, Kyoto, Japan) under ambient conditions. Typical AFM probes (curvature radius, <20 nm) mounted on cantilevers (200 μm) with spring constant of 0.032 N/m were used. Scanned areas (1 Hz) were perfect squares (20 μm $\times$ 20 μm) in which was applied a weak force (<1 nN). AFM images (256 $\times$ 256 lines) were processed with both Shimadzu SPM-9600 AFM analysis software and WSxM Scanning Probe Microscopy Software 4.0 SPMAGE07 (Nanotec Electronica SL, Madrid, Spain). For the purpose of comparison, AMR, MH, and RMS parameters were chosen to evaluate the surface of rotary Ni-Ti files.

All statistical analyses were performed with StatView for Windows 5.0 Software (SAS Institute Inc, Cary, NC). Mean and standard error of the mean values of the AMR, MH, and RMS parameters were calculated. The differences among the groups were

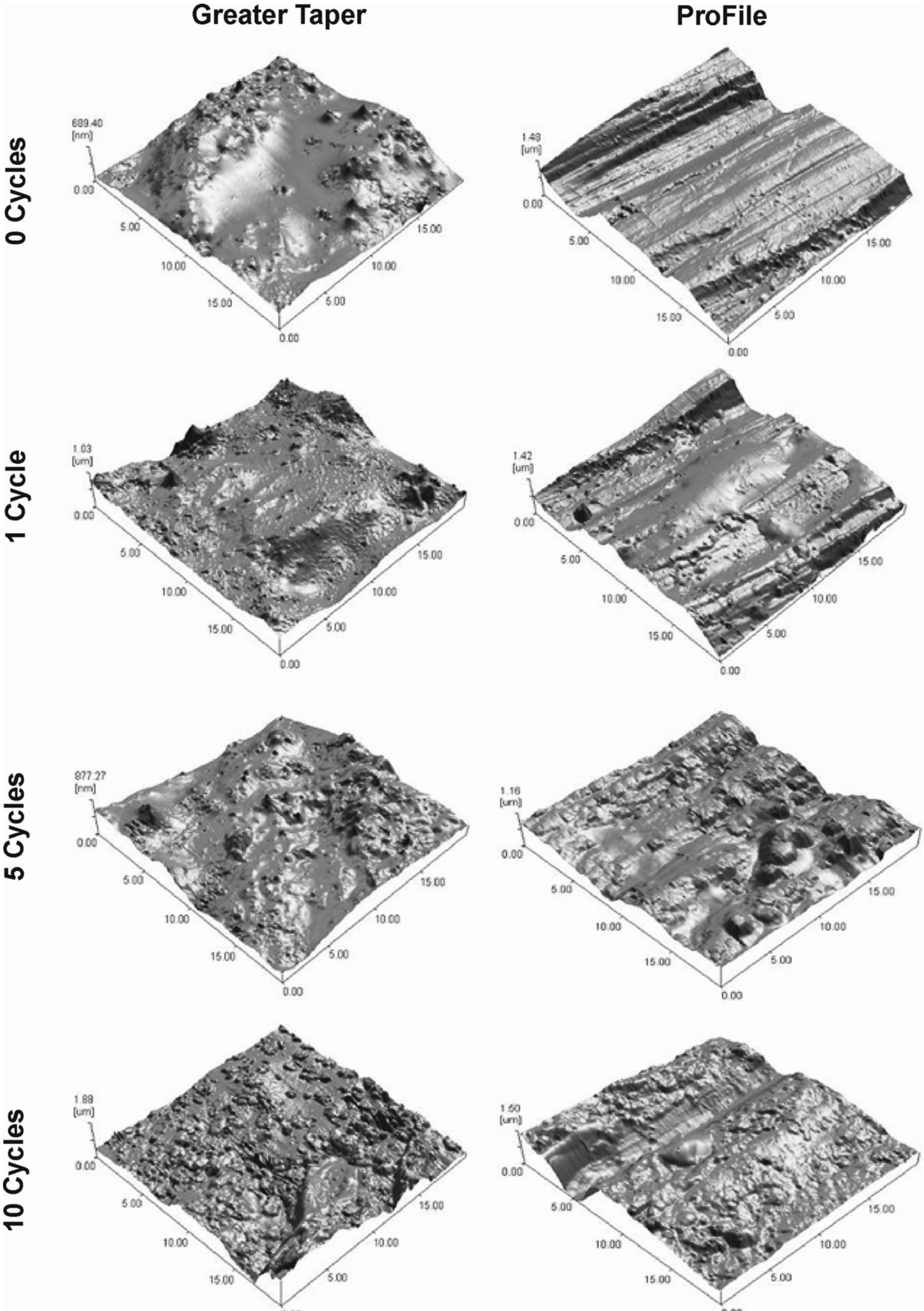

Figure 1. Contact mode AFM 3-dimensional images of typical surface topography of rotary Ni-Ti files from control (0 cycles) and after multiple autoclave cycles (1, 5, and 10). Visual irregularities in the surface topography were observed for both Greater Taper and Profile in all conditions analyzed.

tested by analysis of variance with Tukey test for multiple comparisons and were considered significant when $P < .05$.

Results

Fig. 1 shows contact mode AFM 3-dimensional images of typical surface topography of both Greater Taper and ProFile rotary Ni-Ti files from control (0 cycles) and after multiple autoclave cycles (1, 5, and 10). Visual irregularities in the surface topography were observed for both Greater Taper and Profile in all conditions analyzed.

To investigate any quantitative statistically significant difference in surface topography resulting from autoclave exposures, vertical topographic parameters (AMR, MH, and RMS) were evaluated. Fig. 2 shows vertical topographic parameters of rotary Ni-Ti files expressed as the mean ± standard errors of the mean. The values are expressed in nanometers (nm). Autoclave exposure increased AMR, MH, and RMS parameters for Greater Taper after 10 cycles compared with after 1 cycle and control for the same file sample ($P < .05$). Moreover, Greater Taper showed higher MH and RMS parameters after 10 cycles compared with after 5 cycles for the same file sample ($P < .05$). Concerning ProFile, autoclave exposure increased AMR, MH, and RMS parameters after 5 and 10 cycles compared with control for the same file sample ($P < .05$).

Discussion

Reports concerning effects of autoclave cycles on rotary Ni-Ti files focus mainly on the mechanical properties, resistance to fracture, and cutting efficiency (4–7). However, when assessing quality of rotary Ni-Ti files, the surface character must be taken account, because this determines the corrosion resistance of such instruments under a broad range of conditions. Although scanning electron microscopy has long been the standard means used for surface characterization of rotary Ni-Ti files, AFM was selected for the present study because it is a sensitive and reliable technique that offers a suitable means for acquisition of qualitative and quantitative data concerning surface topography of rotary Ni-Ti files.

As observed in AFM 3-dimensional images for control, surface of rotary Ni-Ti files showed irregularities in topography caused by the manufacturing process (Fig. 1). To evaluate whether depth of irregularities increases after multiple autoclave cycles, 3 different vertical topographic parameters (AMR, MH, and RMS) were assessed. In this case, it was found that autoclave was able to increase AMR, MH, and RMS parameters of the surface topography of both Greater Taper and ProFile rotary Ni-Ti files (Fig. 2). It is well-known that increase of surface irregularities is associated with the fracture mechanism of rotary Ni-Ti files during clinical use, especially in curved canals (8–10). Moreover, it has been demonstrated that surface irregularities also substantially affect cutting efficiency and cleaning of such instruments (8, 11, 16). Therefore, our results for topographic amplitude of surface irregularities localized along rotary Ni-Ti files after autoclave exposure might be clinically relevant.

It is interesting to note that number of cycles needed to increase depth of irregularities located along the surface change between different types of rotary Ni-Ti files. Whereas autoclave increased AMR, MH, and RMS parameters of surface topography of ProFile after 5 repeated autoclave cycles when compared with the control, similar finding was observed on Greater Taper surface only after 10 repeated cycles (Fig. 2). Although the nature of these phenomena is not quite clear, this seemingly contradictory result could be explained by the differences in manufacturing process, which might result in rotary Ni-Ti files more susceptible to surface corrosion when compared with others. This is confirmed by studies that demonstrated differences on surface characteristics among endodontic instruments manufactured by using different alloy composition or production method (15, 17).

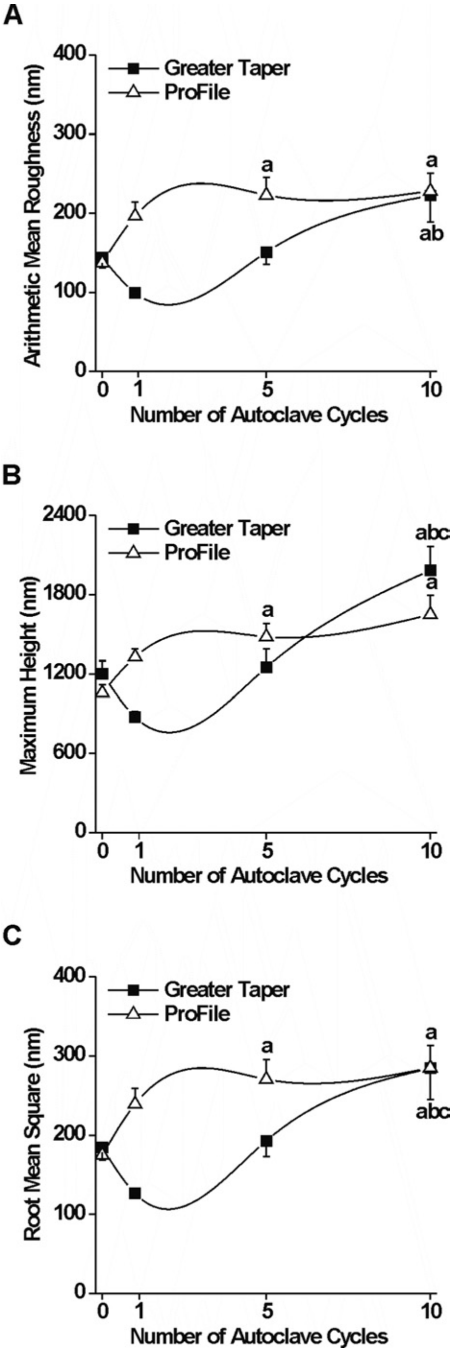

Figure 2. Vertical topographic parameters (*a*) AMR, (*b*) MH, and (*c*) RMS of the rotary Ni-Ti files from control (0 cycles) and after multiple autoclave cycles (1, 5, and 10). (*A*) $P < .05$ when compared with control in the same file; (*B*) $P < .05$ when compared with after 1 cycle in the same file; (*C*) $P < .05$ when compared with after 5 cycles in the same file.

On the other hand, in this study single autoclave sterilization for both Greater Taper and ProFile rotary Ni-Ti files did not increase AMR, MH or RMS parameters of surface topography when compared with the control (Fig. 2), suggesting that single cycle of autoclave is less prejudicial to rotary Ni-Ti files. In fact, although autoclave procedure presents potential to cause structural changes on rotary Ni-Ti files, it has been related that this undesirable effect is only observed after a considerable number of autoclave procedures (5, 6), probably because autoclave has a cumulative effect on structure of the rotary Ni-Ti files, which can result in surface corrosion after an excessive number of cycles. In addition, it is important to mention that clinically several factors, including stress produced during instrumentation and chemical composition of disinfectant solution, also display an adverse effect on surface of rotary Ni-Ti files (12, 16). Such factors probably contribute to accelerate the surface corrosion of rotary Ni-Ti files exposed to autoclave. Therefore, single use or minimal reprocessing of rotary Ni-Ti files might be a safe practice during endodontic therapy.

The surface effect of autoclave on rotary Ni-Ti files was examined by sensitive AFM technique. The results showed that multiple autoclave cycles increase depth of surface irregularities located on active section of rotary Ni-Ti files. Moreover, number of cycles needed to cause this effect on surface topography changed between different types of rotary Ni-Ti files. However, single cycle of autoclave was not prejudicial to any rotary Ni-Ti files evaluated. These findings are promising regarding specification of appropriate number of reprocessing of rotary Ni-Ti files during endodontic practice.

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