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In-depth hardness profiles of Stainless Steel and Ni-Ti endodontic instrument cross-sections by nano-indentation

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Abstract

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Aim To evaluate the in-depth hardness profiles of Stainless Steel (SS) and nickel titanium (Ni-Ti) endodontic instrument cross-sections using a nano-indentation technique.

Methodology Three SS (Reamer, K and Hedström) and three Ni-Ti (ProFile, NRT and Liberator) instruments were studied. After embedding and metallographic preparation the in-depth hardness profiles of instrument cross-sections were measured starting from the cutting surface towards the centre to a depth of 2000 nm using an MTS XP nanoindenter with a Berkovich diamond indenter. The results of hardness measurements of outer (near to cutting edge) and inner locations were statistically analyzed by two-way ANOVA followed by SNK test ($\alpha = 0.05$).

Results For all instrument cross-sections the maximum hardness was obtained at the outer surface followed by hardness attenuation towards the centre of the cross section. The statistical analysis of hardness classified the instruments, for both outer and innermost locations, to the following decreasing order: Reamer > K > Hedström > Profile > NRT shank (without thermal treatment) > NRT tip (with thermal treatment) > Liberator. The maximal hardness, at the outer surface of endodontic instruments, can be attributed to the residual stresses developed due to cutting and thermal effects during the manufacturing process. The increased outer layer hardness may have a beneficial effect on the cutting ability and wear resistance of endodontic instruments.

Conclusions All endodontic instruments had a decrease in hardness towards their centre. This implies that the surface hardness of contemporary endodontic instruments is significantly enhanced by the consequences of manufacturing processes.

Keywords: endodontic instruments, hardness, nano-indentation, Ni-Ti, residual stresses.

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Introduction

Cutting efficiency and wear resistance are among the main requisite properties of endodontic instruments. The former governs the ability of the instrument to cut dentine effectively and the latter reduces the loss of surface material with repeated use.

Instrument hardness and geometry are linked directly with the above properties. Nickel-Titanium (Ni-Ti) instruments cut less well than Stainless Steel (SS) (Tepel *et al.* 1995a, Tepel *et al.* 1995b). This is attributed to the lower hardness of Ni-Ti instruments. Typically Vickers Hardness (HV) values are in the region of $313 \sim 481$ (Kim *et al.* 2005, Alapati *et al.* 2006) as compared to $546 \sim 673$ for SS (Darabara *et al.* 2004). To overcome

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this limitation several surface treatments have been proposed to enhance the surface hardness of Ni-Ti instruments and thus their clinical efficacy (Rapisarda *et al.* 2001, Schäfer 2002a, Schäfer 2002b, Tripi *et al.* 2003, Tripi *et al.* 2002). Although differences between SS and Ni-Ti alloys are associated with their cutting ability, all hardness values have been derived from the inner material of the instruments leaving the outer surface hardness unknown. The matter is further complicated when the residual stresses and metallurgical alterations within the instrument surface, developed during the manufacturing processes, are taken into account. Reamers and K-files are produced by twisting wire blanks inducing a strain hardening effect that leaves the instruments with an intense field of residual stress. Strain or work hardening increases hardness and strength by plastic deformation at temperatures below the recrystallization range. The effect of this stain-hardening is minimal at the centre of the cross section (which remains unstrained during twisting) and highest at the periphery (cutting edge) of the instrument. Hedström, along with all Ni-Ti files, are produced by machine grinding (Miserendino 1991), developing residual stresses at the surface due to thermal and mechanical phenomena during the process (Tricard 1994).

The small size and complicated geometrical features of endodontic instruments prohibit the application of conventional microhardness techniques for determination of outer surface hardness. Therefore a novel technique, for characterizing endodontic instruments, was employed. This nanoindentation or depth-sensing indentation (VanLandingham 2003) allows hardness to be determined at small scales and offers high spatial resolution. Furthermore, due to the small size of the hardness impression, a method has been developed which allows hardness to be derived from the load-displacement data, obtained during a loading-unloading cycle, without the need to image the residual impression (Oliver and Pharr 1992, 2004). Thus, the technique is ideal to characterize small structural features.

The aim of this study was to determine the in-depth hardness profile of cross-sections starting from the outer cutting edge, of representative SS and Ni-Ti endodontic instruments using a nanoindenter.

Materials and methods

Six different instrument types were studied (Table 1). The instrument files were embedded vertically in acrylic resin (Durofix-2, Struers, Copenhagen, Denmark). This resin contains mineral fillers and is recommended for samples with high edge retention requirements (http://www.struers.com). Two specimens of NRT instruments were embedded to investigate the hardness profile for shank (nonthermal treated area) and the tip (thermal treated area). All specimen cross-sections were metallographically ground and polished perpendicular to their longitudinal axis according to recommended preparation procedures (http://www.struers.com). After wet grinding with SiC papers from 220 to 2000 grit size, the SS instruments were polished with diamond powder down to 0.5 μm in a grinding polishing machine (Ecomet III, Buehler Ltd., Lake Bluff, IL, USA). The Ni-Ti instrument cross-sections were ground to a smooth surface using SiC paper up to 320 grit and final polishing was completed using a 9-μm diamond paste (DP paste; Struers) and a solution of 70 mL colloidal silica (OP-S suspension solution; Struers) and 30 mL H_2O_2. All specimens were ultrasonically cleaned (Ultramatic 150; Gunter Jaschke, Freiburg, Germany) in a distilled water bath for 3 min.

The in depth hardness profiles were measured on the instrument cross-sections from the cutting surface (250 nm from the interface with the embedding material) to a depth of 2000 nm, using an MTS XP nanoindenter (Nano Instruments, TN, USA) with a Berkovich diamond indenter with a 50 nN and 0.02 nm resolution in load and displacement respec-

Table 1 Commercial names or instrument type, ISO number and taper, alloy type, Lot numbers and manufacturers

Commercial names/file type	ISO no./taper	Alloy	Lot number	Manufacturer
Reamer	25	AISI 304[a]	0002712	MicroMega Besancon, France
K-file	25	AISI 304[a]	313696	Maillefer Ballaigues, Switzerland
Hedström	25	AISI 304[a]	337457	Maillefer Ballaigues, Switzerland
Profile	25/0.04	Ni-Ti	041006011	Dentsply, TulsaDental, Johnson City, TN
NRT (thermal treated)	25/0.04	Ni-Ti	5040677600	Mani Inc., Tochigi-Ken, Japan
Liberator	25/0.04	Ni-Ti	013P0602	Miltex Inc, York, PA

[a]As determined previously (Darabara *et al.*, 2004).

tively. The continuous stiffness measurement (CSM) technique (Oliver & Pharr 2004) was used, whereby the stiffness is measured continuously during the indentation, achieved by imposing an oscillating force on the indenter while monitoring its displacement response. This technique allows the elastic modulus or hardness to be recorded continuously as a function of depth and is therefore considerably advantageous over the traditional method, whereby elastic modulus or hardness is obtained from the upper portion of the unloading curve (Oliver & Pharr 1992) only for a single depth. Fifty indents were made in each specimen up to a maximum displacement of 2000 nm. The indents were at least 0.5 μm away one from the other to avoid the influence of residual stresses from adjacent impressions.

The loading cycle begun once thermal drift was below 0.05 nm s^{-1}. Hardness (H) was determined using the mathematical formula:

$$H = \frac{P}{A}$$

where P = load and A = contact area. The contact area was determined using the methodology introduced by Oliver & Pharr (1992). The hardness data were transformed from GPa to Vickers (VHN) applying the formula HV = 101.96 GPa (http://www.gordonengland.co.uk/hardness/hvconv.htm). The raw data recorded for each in depth profile were smoothed by the Loess mathematical formula, applying a local smoothing technique with tricube weighting and linear regression functions (SigmaPlot, Systat Software, Inc. San Jose, CA, USA). For each curve the hardness values of the outer (nearest to cutting edge) and inner locations were calculated. The percentage decrease between outer and inner locations was calculated according to the following formula:

$$\text{Percentage decrease} = \frac{H_{outer} - H_{inner}}{H_{outer}} * 100\%$$

The hardness data measurements were statistically analyzed by two-way ANOVA using file type and location as discriminating variables. Significant differences among mean values were identified by SNK *post hoc* test at 95% confidence level.

Results

Representative in-depth hardness profiles are presented for each instrument in Fig. 1. Reamer, K and Hedström instruments depicted a rather linear in-depth profile. By contrast, Ni-Ti instruments had a nonlinear profile up to approximately 1000 nm which became linear with increasing depth. However, this transition was not so clear for the Liberator.

The results of statistical analysis are presented in Table 2. In all cases SS instruments were significantly harder than NiTi instruments. Also, in all cases the highest hardness values were observed at the outer surface followed by hardness attenuation towards the centre of the cross-section.

Reamers had the highest VHN for the outer surface (820) and Liberator had the lowest (402). For the innermost region Reamers and K-files exhibited the highest values (686 and 698 respectively) and the two types of NRT, shank and tip, the lowest (332 and 338 respectively).

Discussion

All instruments were chosen to represent material and manufacturing differences. All SS files were manufactured from the same alloy AISI 304 (Darabara *et al.* 2004) but represented differences in the manufacturing process. Reamers are manufactured from twisting of blanks with triangular cross section while K-files from rectangular ones. Additionally, a K-file has double the number of twists as a Reamer of the same size and as a consequence has approximately double the work-hardening (Miserendino 1991). A Hedström file is produced from the same alloy but with a machine grinding method. All Ni-Ti instruments are produced by machine grinding but there are manufacturing and geometrical differences among them. ProFile and NRT are typical Ni-Ti instruments with surface fluting. However, the first 5 mm from the cutting tip of NRT files have been thermally treated for 30 min at 430 °C enhancing its fatigue resistance (Zinelis *et al.* 2007). The effect of thermal treatment on fatigue resistance has been attributed to metallurgical alterations during the thermal treatment process. Liberator is a recently introduced instrument without fluting.

This study revealed that hardness at the outer surface is significantly higher compared to the inner material, a finding that was common for all instruments tested (Table 2). This behaviour is a consequence of the manufacturing process. This is more complex in machine grinding as two mechanisms with antagonistic effects are implicated (Fig. 2). Mechanical deformation-dependent residual stresses show a compressive nature attributed to the fact that the surface layers of the material undergo some kind of

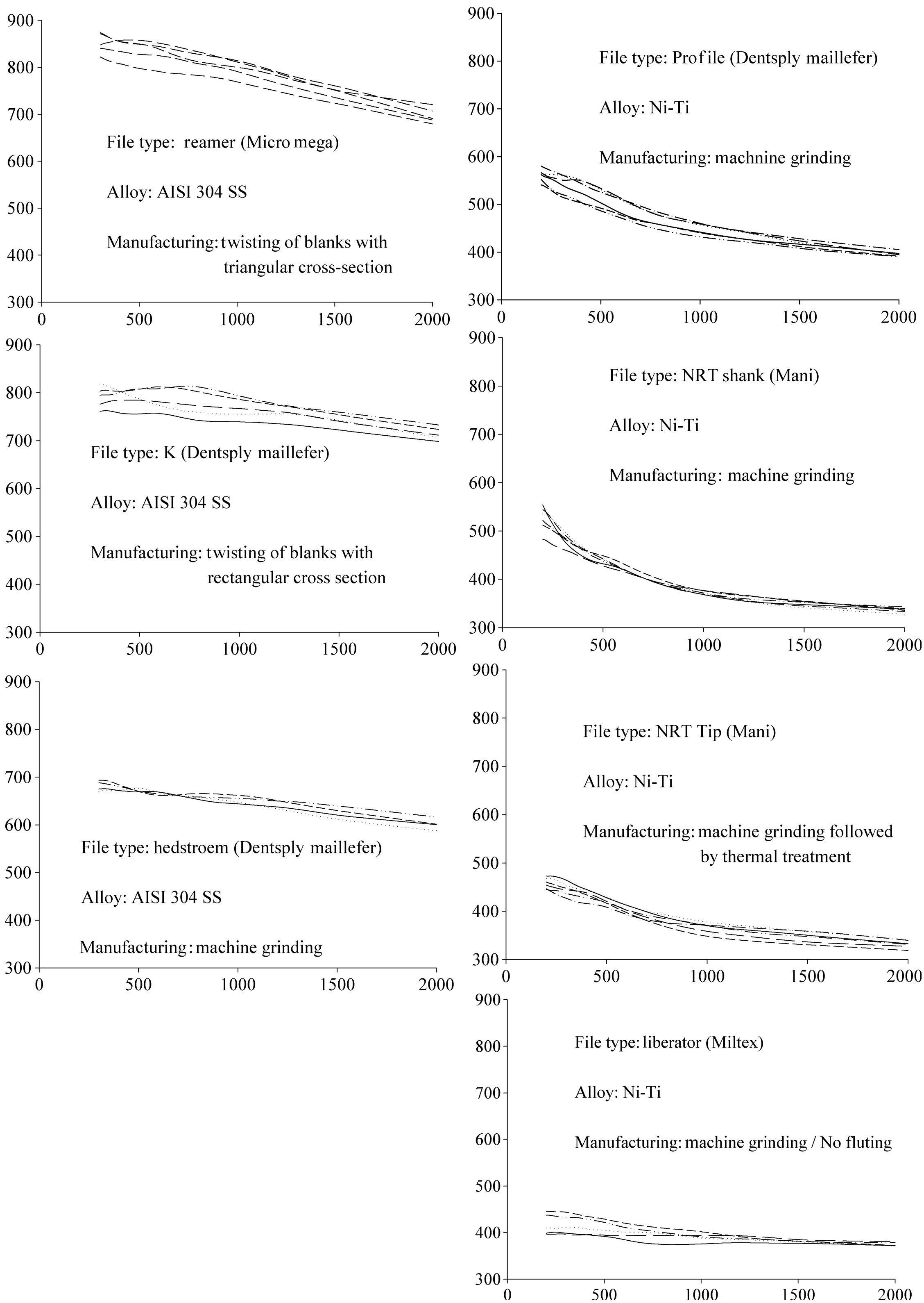
File type: reamer (Micro mega)
Alloy: AISI 304 SS
Manufacturing: twisting of blanks with triangular cross-section
File type: Profile (Dentsply maillefer)
Alloy: Ni-Ti
Manufacturing: machnine grinding
File type: K (Dentsply maillefer)
Alloy: AISI 304 SS
Manufacturing: twisting of blanks with rectangular cross section
File type: NRT shank (Mani)
Alloy: Ni-Ti
Manufacturing: machine grinding
File type: hedstroem (Dentsply maillefer)
Alloy: AISI 304 SS
Manufacturing: machine grinding
File type: NRT Tip (Mani)
Alloy: Ni-Ti
Manufacturing: machine grinding followed by thermal treatment
File type: liberator (Miltex)
Alloy: Ni-Ti
Manufacturing: machine grinding / No fluting
900
800
700
600
500
400
300
0
500
1000
1500
2000

Figure 1 In-depth hardness profiles of endodontic instruments. X axis is the distance in nm from the outer surface towards the centre of the instrument and y axis is hardness in Vickers units. All the graphs have the same x-y scaling to facilitate comparison. Only five scans are presented for each file for the sake of clarity. SS files are presented in the left column and Ni-Ti files in the right one. SS, Stainless Steel.

Table 2 Mean values and standard deviations of hardness values presented in decreasing order, determined after nanoindentation in outer (near to cutting edge) and inner (2000 nm below the cutting edge) for all instruments

Instrument	Outer	Inner	Percentage difference between outer and inner surface
Reamer	820 ± 34^{a1}	686 ± 23^{b1}	16
K-files	765 ± 28^{a2}	698 ± 20^{b2}	9
Hedstroem	673 ± 40^{a3}	605 ± 24^{b3}	10
Profile	540 ± 25^{a4}	399 ± 9^{b4}	26
NRT shank	465 ± 44^{a5}	338 ± 6^{b5}	27
NRT tip	445 ± 23^{a6}	332 ± 9^{b5}	25
Liberator	402 ± 19^{a7}	375 ± 8^{b6}	7

Letter superscripts denote significant differences between locations while numerical superscripts demonstrate significant differences among instruments. Mean values with the same superscripts did not show significant differences ($P > 0.05$).

compaction. By contrast, in the case of thermally-developed residual stresses there is a change in volume in the surface layer of the component when it is thermally treated. Surface layers, which were initially expanded due to heating, contract after heat removal. However, the material bulk resists this contraction, causing the surface layer to go into tension and the bulk into compression. The residual stresses induced by the conjoint action of thermal and mechanical deformation are representative of those induced by machine grinding and of course the two phenomena cannot be disassociated.

As stated above, K-files have double work-hardening compared with the Reamers and thus it is expected that K-files should have higher hardness. However, the Reamer had significantly higher hardness than the K-files for outer and inner locations, a finding which is in accordance with the results of a previous study for the bulk hardness of these specific instruments (Darabara *et al.* 2004). An explanation for this discrepancy might be the possible difference of the extent of work hardening during cold drawing, a process that is used for manufacturing wires. It is expected that the extent of work hardening could vary between different manufacturers. Of course, such factors as the extent of cold drawing for wire manufacturing, intermediate annealing process, and other manufacturing details generally described by the term 'thermomechanical history' are proprietary and remain undisclosed by the manufacturers. This makes any comparison among different materials difficult. Additional information for wires and files manufacturing processes are available in the dental literature (Miserendino 1991, Thompson 2000). The higher hardness found for the outer surface of Hedström files is associated with the superficial residual stress field developed during machining. This is a typical finding after machining of SS (M'Saoubi *et al.* 1999, Warren and Guo 2006).

In-depth hardness profiles of Ni-Ti instruments demonstrate a nonlinear profile up to approximately 1000 nm which became linear for the subsequent region, although this was not the case for Liberator. ProFiles had the highest hardness value for the outer layer followed by the NRT without thermal treatment (shank), NRT with thermal treatment (tip) and Liberator. The higher hardness found at the outer locations are associated with superficial compressive residual stresses that develop during the grinding process. Another interesting finding is the substantial differences between the geometrical and manufacturing processes of the instruments. ProFiles demonstrated the highest hardness at the outer surfaces a finding that may be attributed to the flute geometry and/or to the fact that ProFiles are manufactured from wires with a larger extent of work hardening. The latter is also associated with the significantly higher hardness of ProFile for the inner region compared to the rest Ni-Ti instruments. The thermal-treated tip of the NRT instruments had the lowest hardness values only for the outer location implying that the selected thermal treatment relieved only a fraction of the superficial residual stresses. The Liberator instrument demonstrated the lowest hardness at the outer location, along with the lowest percentage difference in hardness between outer and inner, implying that the creation of the surface fluting was implicated with the development of a more intense superficial layer of residual stresses. The hardness increase between inner and outer locations of Liberator file should be attributed to the surface grinding for taper production and to the limiting twisting during the production process.

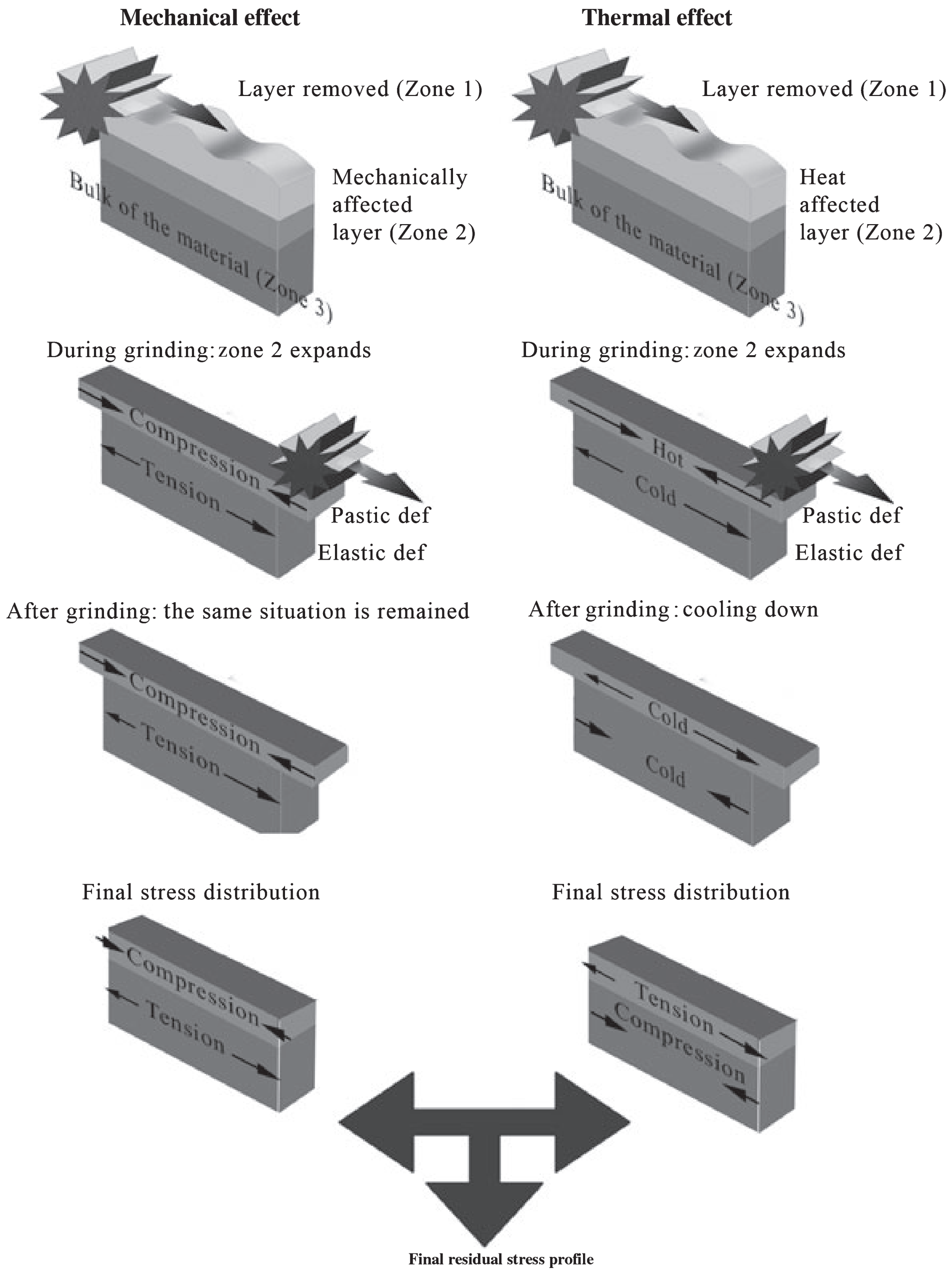

Figure 2 Residual stress profile developed in ground surfaces by overlapping of the mechanical (left) and thermal (right) effects. Mechanical effect (left): Three distinct zones can be distinguished. Zone 1 represents the layer being removed. Zone 2 is the mechanically affected zone and zone 3 represents the bulk of the material. During grinding, zone 2 undergoes plastic deformation which is limited by the unstrained zone 3. After the completion of surface grinding, zone 2 is left in compression while zone 3 is in tension. Thermal effect (right): Again in this case zone 1 and zone 3 represent the removed layer and bulk of the material respectively. Zone 2 is the *Heat Affected Zone* (HAZ). During grinding the heated zone 2 expands in length due to thermal expansion buts once again this expansion is constrained by the bulk of material (zone 3) which remains at room temperatures leaving the surface under compression. After the grinding process the workpiece starts to cool down, reversing the distribution and resulting in tension for zone 2 and compression for the bulk of the material. Finally the real profile of residual stresses is the sum of mechanical and thermal effects. [Redrawn from Tricard 1994].

A limitation of this study is that the first recorded hardness values were taken from a depth around 250 nm below the cutting edge of the instruments. The origin of this limitation is associated with the edge developed at the interface between the specimen and the embedding material during the metallographic preparation. Epoxy and acrylic resins abrade to a lower general level than the alloys and ceramics resulting in the rounding of the specimen edge to match the differences in level (Samuels 1985). Although the most appropriate embedding materials for edge retention was used in this study this complication was not completely overcome. This limitation masked the hardness properties at the outermost (and the most important) region, which is related to the cutting efficiency and the wear resistance of the instrument tested. Unfortunately, there are no reports in literature for the hardness at the outermost region nor for the residual stresses developed during the manufacturing process and thus only assumptions can be made about the hardness properties of this outermost layer. Besides hardness measurements with the nano indentations technique cannot be made directly on the surface of the instrument due to the intense surface roughness. Reamers and K-files are produced by twisting and thus the maximum work hardening is developed at the periphery. Therefore, it is logical to assume that the hardness will increase up to the external surface. This matter is much more complicated for the instruments produced by grinding, wherein the antagonistic effect of tensile stresses due to thermal effect and compression stresses due to grinding coexist at the same region. The experimental data presented in this study could be used for finite element analysis to determine residual effects of the manufacturing process at the instrument subsurface. Analysis of residual stresses with other analytical techniques (Wyatt and Berry 2006) is also an interesting field for further research.

The results of this study have clinical significance since they relate to the cutting ability and wear resistance of endodontic instruments. Although residual stresses are developed due to side effects of manufacturing processes they remain completely unknown. The increased hardness of the outermost layers has a beneficial effect in cutting efficiency and wear resistance of endodontic instruments. Nevertheless, both properties are also dependent on the geometrical features of the instrument. The results of this study clearly show that the comparison of the aforementioned properties for different endodontic instruments cannot be related to the bulk hardness measured by microhardness techniques. Surface properties of endodontic instruments are changed as a result of manufacturing processes. This opens a very interesting field for further research to optimise their surface properties according to clinical demands.

Conclusion

All endodontic instruments (SS and Ni-Ti) decreased in hardness towards their centre. All SS instrument demonstrated higher hardness values compared to Ni-Ti for inner and outer locations.

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