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CHARACTERIZATION OF NEXT-GENERATION NICKEL-TITANIUM ROTARY ENDODONTIC INSTRUMENTS

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ABSTRACT

Background: Next-generation rotary endodontic instruments (GT® Series X™) with improved clinical performance have been fabricated from special superelastic NiTi wire with a nanoscale martensitic structure. Last year we reported Vickers hardness measurements and SEM images of as-received and clinically used GT® Series X™ instruments. In this manuscript, we report characterization of the nickel-titanium instruments, using Micro-X-ray diffraction and differential scanning calorimetry (DSC). Results: X-ray diffraction shows evidence of the martensitic structure that accounts for the much higher Vickers hardness and superior wear resistance of the next-generation NiTi instruments, compared to previous instruments. DSC analyses show that fabrication of these instruments from the starting special superelastic wire has little effect on NiTi transformations and that most of the NiTi microstructure does not undergo transformation during heating to 120°C. Conclusions: Micro-X-ray diffraction and DSC provide important characterization information for these instruments, but TEM is needed for detailed insight the complex martensitic structure.

INTRODUCTION

Following the pioneering research by Walia et al[1] on nickel-titanium hand instruments for root canal therapy, nickel-titanium rotary endodontic instruments used with a slow-speed dental handpiece have achieved widespread popularity since the original ProFile® instrument was marketed by Tulsa Dental (now Dentsply Tulsa Dental Specialties) nearly twenty years ago. The low elastic modulus and pseudoelastic character of the near-equiatomic NiTi alloy used with the commercial instruments allows them to negotiate curved root canals with facility.[2] Nevertheless, occasional clinical fracture of the conventional NiTi rotary instruments has been reported,[3] which may be due to cyclic loading or a single episode of sudden overload.[4]

While there have been ingenious surface modification strategies[5-7] to improve the properties and clinical performance of the conventional NiTi instruments, recent notable advances for the rotary NiTi instruments have been the consequence of a paradigm shift from the

austenitic structure for the starting NiTi wire blanks from which conventional instruments[2] are machined. The seminal innovation was the introduction of a special M-Wire[8] with a nanoscale martensitic structure, processed by a proprietary thermomechanical technique. Dentsply Tulsa Dental Specialties subsequently marketed GT® Series X™ instruments manufactured from M-Wire, and our research group previously presented Vickers hardness measurements and scanning electron microscope (SEM) observations of as-received and clinically used GT® Series X™ instruments.[9] The decreased wear of these instruments observed with the SEM, compared to that for conventional NiTi rotary instruments, was attributed to their higher hardness from the work-hardened martensitic structure. The present complementary study summarizes our metallurgical characterization of the used and as-received GT® Series X™ instruments, with comparisons to previous observations of the M-Wire blanks,[8] using Micro-X-ray diffraction (Micro-XRD) and differential scanning calorimetry (DSC).

EXPERIMENTAL PROCEDURES

New GT® Series X™ instruments were provided by Dentsply Tulsa Dental Specialties, and GT® Series X™ instruments and instruments that had experienced 7 – 8 clinical uses were obtained from the Dental Faculty Practice of the College of Dentistry at The Ohio State University.[9]

Micro-XRD analyses[10,11] were performed (Rint-2000, Rigaku) were performed near the instrument tip and at 3 mm and 6 mm distances from the tip (CuKα radiation, 40 kV tube voltage and 300 mA tube current), using an analysis area of approximately 100 μm. Specimen temperatures of 5°, 25°, 37° and 60°C were utilized for the initial experiments, and subsequent Micro-XRD analyses were performed at 25°C.

For DSC analyses, 4 - 5 mm length segments that included the instrument tip and two adjoining regions were cut from water-cooled instruments (Isomet, Buehler). Each test specimen (one or more segments) was placed in an open aluminum pan, and an empty aluminum pan served as the inert control specimen. Conventional DSC was performed (Model Q100, TA Instruments), and specimens were first cooled from room temperature to -80°C, then heated to 120°C, and subsequently cooled back to -80°C, using a heating or cooling rate of 10°C/min. Dry nitrogen was used as the purge gas. Computer software (TA Universal Analysis 2000) with the DSC apparatus was used to obtain the onset temperatures for phase transformations and the associated enthalpy changes (ΔH). Interpretations of the DSC plots followed a previous study of conventional endodontic instruments.[12]

RESULTS

Figure 1 shows the Micro-XRD patterns at 5°, 25°, 37° and 60°C for the region 3 mm from the tip of an as-received GT® Series X™ rotary instrument, and it can be seen that there were minimal differences for these temperatures. Figures 2 and 3 present the Micro-XRD patterns at 25°C near the tip region and at distances of 3 mm and 6 mm from the tip for new and used GT® Series X™ rotary instruments, respectively. A variety of sizes and tapers for the GT® Series X™ rotary instruments were examined, and there were no evident differences in Micro-XRD patterns for new and used instruments.

Four peaks were generally observed (near 40°, between 42° and 43°, near 62°, and near 78°), and were indexed to martensite and austenite peaks[13] from the ICDD standards for and to published[14] peak positions for R-phase. The intensity of the x-ray diffraction peaks was generally lowest near the tip region, compared to positions 3 mm and 6 mm from the tip. For the weak peak near 40°, the 020 plane for martensite is the best match; the 110 plane for martensite may be

possible if there is extreme preferred orientation. The large peak between 42° and 43° can have contributions from the $1\bar{1}1$ and 002 (less likely) planes for martensite, the 110 plane for austenite, and the 112 and 300 planes for R-phase. The peak near 62° can be indexed to the 200 plane of austenite and the 222 plane of R-phase. The peak near 78° can be indexed to the 211 plane of austenite and and the 412 and 330 planes of R-phase.

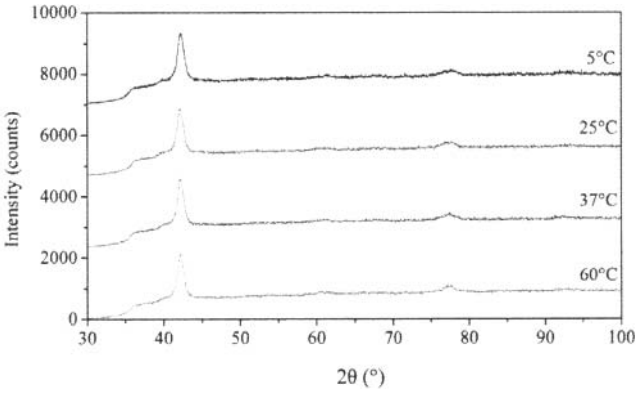

Figure 1. Micro-XRD patterns at four temperatures for site 3 mm from tip region of representative as-received GT® Series X™ instrument.

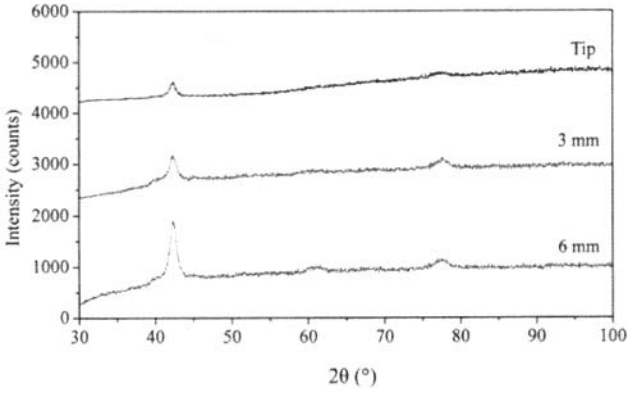

Figure 2. Micro-XRD patterns at 25°C from 3 sites on representative as-received GT® Series X™ instrument.

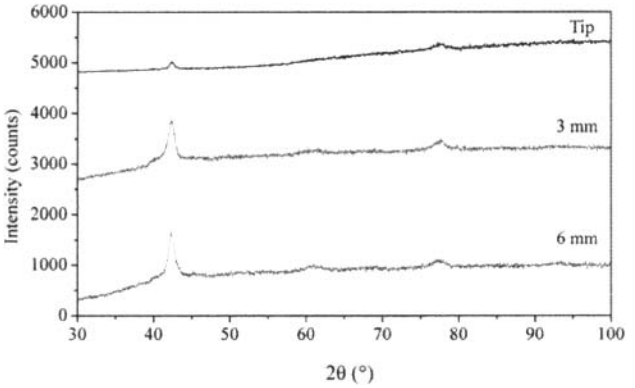

Figure 3. Micro-XRD patterns at 25°C from 3 sites on representative clinically used GT® Series X™ instrument.

Figure 4 presents the DSC heating (lower) and cooling (upper) plots for the segment that contained the tip of a representative used GT® Series X™ instrument. Figure 5 shows the DSC plot for the segment that contained the tip of another used GT® Series X™ instrument of much smaller size. The tip region is of interest because this portion of the rotary instrument experiences more work hardening during the manufacturing process and more mechanical forces during clinical use.

The endothermic peak on the heating plot corresponds to the transformation from martensite to austenite, and the exothermic peak on the cooling plot to the reverse transformation from austenite to martensite.[12] The onset temperatures for the transformations are shown, along with the enthalpy changes (ΔH). The DSC plots indicated that at body temperature (37°C) the instruments should be a mixture of martensite and austenite, since neither the heating nor the cooling transformation has been completed. In these figures conventional DSC analyses were unable to distinctly resolve the R-phase, which can form as an intermediate structure during transformation between martensite and austenite, although evidence of R-phase transformations was observed for other specimens.

Values of ΔH for the forward and reverse transformations between martensite and austenite were typically between 1 J/g and 2 J/g, as shown in Figure 4 for the segment containing the tip of a representative used GT® Series X™ instrument. Similar DSC plots were found for specimens containing all three segments from a given new or used instrument. The full range of ΔH for all instruments was about 0.4 J/g to 8 J/g. The DSC plot for a used GT® Series X™ instrument segment containing the tip region, having ΔH of about 8 J/g for the heating transformation from martensite to austenite is portrayed in Figure 5. Interestingly, the enthalpy change for the cooling transformation from austenite to martensite in this specimen was about 1.8 J/g. For most instruments analyzed the austenite-finish (A_f) temperature was close to 50°C, and varied little (generally less than 5°C) with size and taper, and for new and used instruments.

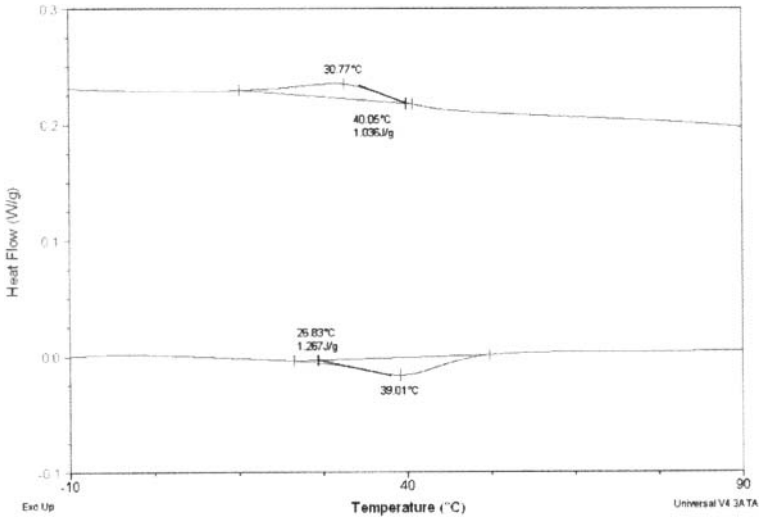

Figure 4. DSC plots segment with tip of representative used GT® Series X™ instrument.

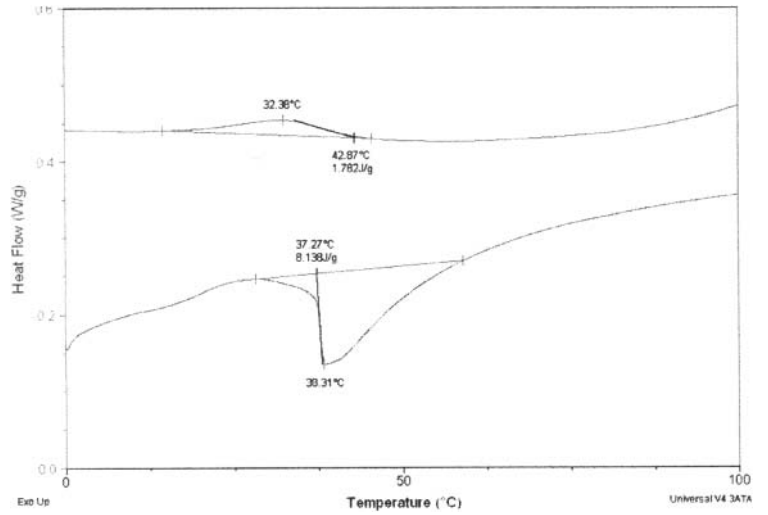

Figure 5. DSC plots for segment with tip of different used GT® Series X™ instrument.

DISCUSSION

Comparison of the DSC plots for the GT® Series X™ instruments obtained in the present study with our previous results for the starting M-Wire blanks[8] shows that there are not noteworthy differences in the A_f temperatures. However, ΔH values for the transformation between martensite and austenite are generally higher for the wire blanks (more than approximately 3 J/g), and the two-step heating transformation involving the R-phase was evident.[8] These differences are attributed to the additional work hardening of the nickel-titanium alloy when the instruments are machined, but transmission electron microscopy is required to elucidate differences in the complex ultrastructures. Figure 6 presents the nanoscale details for the martensitic structure of the starting M-Wire blanks.

Figure 6. Bright-field transmission electron microscope photograph (STEM mode) of an M-Wire specimen. [From the study reported in Reference 8.]

Our previous transmission electron microcopy (TEM) observations revealed that the NiTi wire used for conventional rotary instruments had a submicron austenitic structure, in contrast to the martensitic structure of M-Wire which had a coarser architectural scale.[8] The processing of both the conventional pseudoelastic wire and the M-Wire resulted in substantial residual permanent deformation effects and much lower values of ΔH for the transformation between martensite and austenite[8] than occur for the shape memory and pseudoelastic orthodontic wires where ΔH often exceeds 10 J/g[15]. Thus it can be concluded that DSC analyses, while informative and yielding the clinically desirable A_f temperatures, only provide partial information about the NiTi microstructures, i.e., about the phases that are capable of undergoing transformation. Stable martensite in the starting NiTi wire blanks and the subsequently machined instruments will not be detected. Nonetheless, the conventional DSC analyses illuminate a subtle change in the character of the NiTi transformations in the machined instruments compared to the wire blanks, with the R-phase transformation no longer always being strongly evident for the instruments.

While Micro-XRD is a highly convenient complementary method to obtain details about the NiTi phases in the starting wire blanks and machined instruments, there are important concerns. X-ray diffraction peaks are decreased in intensity because of the inevitable work hardening during wire processing and subsequent machining of the rotary instruments, and preferred orientation can alter relative peak intensities considerably from those expected from the ICDD standards and the published peaks for R-phase.[16] As previously noted, unambiguous peak assignment to specific phases is difficult because multiple peaks for the NiTi phases can occur near the observed peak positions.[8] Given the nanostructure character of the NiTi microstructures, high-resolution TEM with complementary electron diffraction is the best method for characterizing the complex phase relationships in these materials.

In closing, our present research should be regarded as an initial materials science-oriented characterization of these important NiTi endodontic instruments. A second generation of instruments has been introduced by manufacturers, and new detailed materials science studies of the relationships of their microstructures to mechanical properties and clinical performance are needed. A particularly interesting development has been introduction of the Twisted File™ (SybronEndo, Orange, CA), where the NiTi wire blank is twisted to obtain the desired fluted configuration for the rotary instrument rather than being machined or ground and is claimed to have the R-phase structure. Potentially, avoiding the necessity of machining the rotary instruments from wire blanks should result in less likelihood for clinical instrument fracture at sites of machining damage.[4] Another area of practical importance and future study is the use of heat treatments to obtain optimum microstructures of the rotary NiTi instruments for superior clinical performance,[17] appreciating that the basic metallurgy of these NiTi alloys is complex.[18]

CONCLUSIONS

While there was little difference in austenite-finish (Af) temperatures for the GT® Series X™ instruments and starting M-Wire blanks, machining of the instruments resulted in substantial decrease in the enthalpy change (ΔH) for the transformation between martensite and austenite as well as much less evidence from the conventional DSC plots of R-phase involvement in the transformations. These changes are attributed to work hardening during machining of the instruments from wire blanks.

Results from the present study emphasizes that DSC only detects that portion of the NiTi microstructure which undergoes phase transformation, and the microstructure of the GT® Series X™ instruments is dominated by stable martensite, which results in much lower values of ΔH than occurs for transformations between martensite and austenite in shape memory and pseudoelastic orthodontic wires. Moreover, while Micro-XRD does provide useful information about the NiTi phases in these rotary instruments, interpretation is hindered by multiple phases associated with individual peaks and effects of preferred orientation.

Future studies on currently marketed and forthcoming NiTi rotary instruments using TEM with electron diffraction are recommended, along with investigations of heat treatments that might further improve the properties and clinical performance of these instruments.

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