Phase Transformation Behavior and Mechanical Properties of Thermomechanically Treated K3XF Nickel-Titanium Instruments

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Abstract

Introduction: The bending and torsional properties of thermomechanically treated K3XF (SybronEndo, Orange, CA) nickel-titanium instruments in relation to their phase transformation behavior were evaluated. **Methods:** NiTi instruments K3 (SybronEndo) and K3XF, both in sizes 25/.04 and 40/.04, were examined by differential scanning calorimetry and X-ray diffraction. The metal composition was determined by scanning electron microscopy with X-ray energy-dispersive spectrometric analyses. The bending property of K3 and K3XF instruments was measured in a cantilever-bending test with a maximum deflection of 4.00 mm. A torsional test of the instruments was evaluated according to the American National Standards Institute/American Dental Association Specification No. 28. **Results:** K3 and K3XF instruments had approximately the same chemical composition with a nickel content of 48–49 atomic %. The differential scanning calorimetry analyses showed that each segment of the K3XF instruments (24.89°C ± 1.98°C) had a higher austenite finish temperature than the K3 instruments (17.63°C ± 1.76°C) ($P < .05$). The bending load values were significantly lower for K3XF than for K3 in the superelastic ranges ($P < .05$). There was no statistically significant difference between K3 and K3XF in the maximum torque or maximum angular deflection before failure. The torque at fracture values of K3 and K3XF increased significantly with the diameter ($P < .05$). **Conclusions:** K3XF exhibited different phase transformation behavior and flexibility when compared with K3, which may be attributed to the special heat treatment history of K3XF instruments. *(J Endod 2013;39:919–923)*

Key Words

Bending, K3XF, nickel-titanium instrument, phase transformation, thermomechanical treatment, torsion

The development of nitinol, an equiatomic alloy composed of nickel and titanium, has proved to be a significant advancement in the manufacture of endodontic instruments. Nickel-titanium (NiTi) is called an exotic metal because it does not conform to the typical rules of metallurgy. NiTi alloy has special characteristics of superelasticity and shape memory (1). Superelasticity is associated with the occurrence of a phase transformation of the alloy upon application of stress above a critical level, which takes place when the ambient temperature is above the so-called austenite finish temperature of the material. This stress-induced martensitic transformation reverses spontaneously upon release of the stress; the material then returns to its original shape and size (2). This reversible thermoelastic martensitic transformation is the main reason for increased flexibility of NiTi instruments over traditional stainless steel ones, which facilitates instrumentation of curved root canals (3). Despite their increasing popularity, a concern with the use of NiTi rotary instruments is the possibility of unexpected separation during use (4–6). Two mechanisms that may lead to NiTi rotary fracture, cyclic fatigue and torsional overloading, have been described (7, 8).

Instrument fracture is a complex, multifactorial event, and many factors (9, 10), such as the operator, root canal anatomy, and instrument properties, influence the fracture risk. Increasing the resistance to file separation has been the main goal of manufacturers in developing the latest NiTi rotary instruments, aiming at improving safety through innovative design and manufacturing processes (11–15). Thermal treatment of NiTi alloys, such as M-Wire (Dentsply Tulsa Dental Specialties, Tulsa, OK) (13, 14, 16), R-phase wire (SybronEndo, Orange, CA) (11, 17), and controlled memory wire (CM Wire; DS Dental, Johnson City, TN) (18–20), has been used to optimize the mechanical properties of the files. Thermomechanical processing is a frequently used method to optimize the microstructure and transformation behavior of NiTi alloys, which, in turn, have great influence on the reliability and mechanical properties of NiTi files (13, 17–20). Recently, a special thermal process was introduced to NiTi files after the grinding process was completed. Theoretically, the main advantage gained by a specific heat treatment is not only to improve the flexibility and strength of the file but also at the same time by modifying the crystalline structure of the alloy to accommodate some of the internal stress caused

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by the grinding process. Therefore, the new technique could eliminate many drawbacks of the grinding process and produce instruments with superior mechanical properties. The manufacturer claims that K3XF (SybronEndo) provides clinicians with the basic features of the original K3 (SybronEndo) plus an extraordinary new level of flexibility and resistance to cyclic fatigue with the proprietary R-phase technology. These claims of the manufacturers have not been adequately tested by independent research. Three recent studies (21–23) showed that K3XF instruments have superior fatigue resistance compared with conventional superelastic NiTi instruments. However, the fundamental mechanism for this improved performance is unknown. Because the microstructure and phase transformation behavior determine the mechanical properties of NiTi alloys, the study of the phase transformations could provide significant information for the new K3XF instruments. Furthermore, the relationship between thermal behavior and mechanical properties of K3XF instruments has not been investigated sufficiently. Therefore, the hypothesis in the present study is that thermomechanical processing used for K3XF instruments results in significant changes in the phase transformation behavior, flexibility, and torsional resistance compared with K3 instruments manufactured with a traditional process without any heat treatment.

Materials and Methods

Differential Scanning Calorimetry

Instruments K3 (lot 061277639 for size 25/.04 and lot 051274192 for size 40/.04) and K3XF (lot 021278923 for size 25/.04 and lot 021274686 for size 40/.04) (6 files of size 25/.04 and 40/.04 each) were evaluated. Twelve specimens were carefully cut from each instrument using a water-cooled, low-speed diamond saw (Buehler Ltd, Lake Bluff, IL). Each test specimen for differential scanning calorimetry (DSC) analysis consisted of 1–2 segments, each approximately 4–5 mm in length, that were from adjacent portions of the shaft. Each test specimen was placed in an open aluminum pan; no crimped pan top was used to avoid mechanical stresses on the specimens. An empty aluminum pan served as the inert control specimen for the DSC measurements.

The DSC analyses were conducted (PYRIS, Perkin Elmer Diamond Series DSC; PerkinElmer, Shelton, CT) over a temperature ranging from −100°C to 100°C by using the liquid nitrogen cooling accessory to achieve subambient temperatures. The austenitic transformation start and finish points (A_s and A_f) and the martensitic reverse transformation start and finish points (M_s and M_f) were determined by the intersection of an extrapolated baseline and the maximum gradient line of the lambda-type DSC curve. This procedure has been described in detail in previous articles (24, 25).

X-ray Diffraction

X-ray diffraction (X'Pert PRO; PANalytical BV, Almelo, The Netherlands) was performed (26, 27) to identify phases in the instruments and complement the DSC results. Analyses were performed on 6 samples each at room temperature (25°C), with CuKa monochromatic radiation at 40 kV and a tube current of 100 mA. Segments from the shank portions of NiTi instruments were cut from the file using a low-speed, water-cooled diamond saw (Buehler Ltd), and then multiple segments were adhered together and ground by using 2000# SiC sandpaper to obtain a plane. The segments were then cleaned by distilled water and placed side by side on the glass sample holder with an area of approximately 8 × 8 mm^2. The peaks were identified by using the pattern library Powder Diffraction File (PDF release 2004; International Centre for Diffraction Data, Newtown Square, PA) with the MDI Jade5.0 software (Materials Data, Inc, Livermore, CA).

X-ray Energy-dispersive Spectrometric Analyses

Six instruments for each group were measured by scanning electron microscopy (Helios Nanolab 650; FEI, Eindhoven, The Netherlands) with energy-dispersive spectrometric (EDS) analyses. All instruments were ultrasonically cleaned in absolute alcohol before the test. One EDS spectrum was collected from the central region of each specimen at 800× magnification. The quantitative analysis was performed in the nonstandard analysis mode using atomic number correction (ZAF) methods (26).

Bending Resistance Test

The cantilever bending test, according to previous studies (17, 25), was conducted using a universal testing machine (Instron 3365; Instron, Norwood, MA) with a temperature chamber (Instron SFL) and a 5 kN full-scale load cell (accuracy: ±0.4% of reading). Twelve files for each group were tested. After cutting off the handle, each instrument was loaded at 3.0 mm from the tip until a deflection of 4 mm was produced, and the unloading process was then started until the force was reduced to 0 N. The tests were conducted at 37°C in the temperature chamber, and the bending loads at deflections of 0.5 and 3.0 mm in the loading process were recorded.

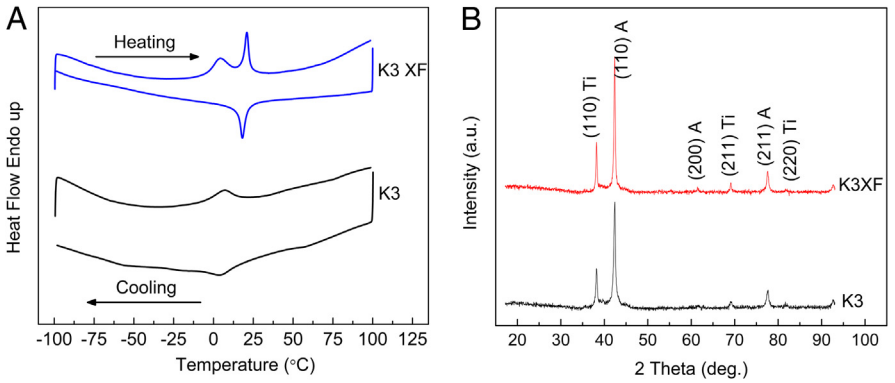

Figure 1. (*A*) Differential scanning calorimetry curves of K3XF and K3 instruments. Heating (*upper*) and cooling (*lower*) curves are shown. (*B*) X-ray diffraction patterns for K3XF and K3 at 25°C, which contain 3 major peaks for the (110), (200), and (211) atomic planes in austenite.