Physical and mechanical characterization and the influence of cyclic loading on the behaviour of nickel-titanium wires employed in the manufacture of rotary endodontic instruments

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Abstract

Bahia MGA, Martins RC, Gonzalez BM, Buono VTL. Physical and mechanical characterization and the influence of cyclic loading on the behaviour of nickel-titanium wires employed in the manufacture of rotary endodontic instruments. *International Endodontic Journal*, **38**, 795–801, 2005.

Aim To analyse the influence of cyclic loading on the mechanical behaviour of nickel-titanium (NiTi) wires employed in the manufacture of ProFile rotary endodontic instruments.

Methodology Nickel-titanium wires, 1.2 mm in diameter, taken from the production line of ProFile rotary endodontic instruments before the final machining step, were tensile-tested to rupture in the as-received condition and after 100 load–unload cycles in the superelastic plateau (4% elongation). The wires were characterized by X-ray energy-dispersive spectroscopy, X-ray diffraction and by differential scanning calorimetry and compared with new size 30, .06 taper ProFile instruments. The fracture surfaces of the wires were observed by scanning electron microscopy.

Results The mechanical properties of the as-received wires, their chemical composition, the phases present and their transformation temperatures were consistent with their final application. Only small changes, which decreased after the first few cycles, took place in the mechanical properties of the cycled wires. The stress at maximum load and the plastic strain at breakage remained the same, while the critical stress for inducing the superelastic behaviour, which is related to the restoring force of the endodontic instruments, decreased by approximately 27%.

Conclusions The mechanical behaviour of the NiTi wires was modified slightly by cyclic tensile loading in the superelastic plateau. As the changes tended towards stabilization, the clinical use of rotary NiTi ProFile instruments does not compromise their superelastic properties until they fracture by fatigue or torsional overload, or are otherwise discarded.

Keywords: cyclic loading, endodontic instruments, fatigue, mechanical properties, nickel-titanium, superelastic wires.

Received 26 April 2004; accepted 7 June 2005

Introduction

Shaping curved and narrow root canals is a challenge, as iatrogenic errors may occur, producing defects such as zips, ledges and canal transportation, which may adversely alter root canal morphology and subsequent prognosis. The introduction of superelastic nickel-titanium (NiTi) alloys in the manufacture of engine-driven endodontic instruments has simplified the preparation of root canal systems. Rotary instrumentation facilitates the maintenance of the original canal shape and the position in space of the apical foramen (Pettiette *et al.* 2001). However, rotating an endodontic instrument during shaping of curved root canals will

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subject it to tensile-compression deformation cycles, eventually leading to fracture by fatigue (Pruett *et al.* 1997, Melo *et al.* 2002).

The main characteristic of the superelastic behaviour exhibited by NiTi alloys is a large recoverable tensile strain (up to 8%, upon unloading), which gives rise to the substantial flexibility of NiTi rotary instruments. This behaviour of NiTi alloys is associated with the stress-induced transformation of the parent β-phase, austenite, with the B2 cubic crystal structure, to martensite, with a monoclinic B19′ structure. Appropriate chemical composition and thermomechanical treatments are required in order that this stress-induced martensitic transformation takes place in NiTi alloys. The martensitic transformation can also occur when the alloy is cooled below a certain temperature (thermal martensite), called martensite start temperature (Ms), and is completed when a lower temperature, martensite finish (Mf), is attained. The reverse transformation, from martensite to austenite during heating, starts at austenite start temperature (As) and finishes at austenite finish (Af). The superelastic behaviour of NiTi alloys takes place when austenite accommodates external stresses by transforming to martensite at a temperature above Af, changing back to austenite as soon as the external stresses are removed (Otsuka & Wayman 1998). The stress required to promote the transformation from austenite to martensite, known as the transformation stress, is an important parameter in the endodontic application of superelastic NiTi alloys, because it defines the restoring force acting on the instrument. In general, lower transformation stresses correspond to lower restoring force, a desired characteristic for curved root canal shaping.

The various processes involved in the production of NiTi rotary endodontic instruments were described in detail by Thompson (2000). Optimized superelastic properties are obtained in NiTi alloys with Ni in excess of 50.5 at.% by cold working, which is accomplished by wire drawing, in the manufacture of the majority of medical and dental applications of this alloy. Cold worked NiTi alloys designed to be superelastic must be heat-treated at relatively low temperatures (around 350 °C), in order to promote partial recovery of the deformed microstructure and the precipitation of the intermediate phase Ti_3Ni_4, which favours the formation of the R-phase, another martensitic phase whose presence in the alloy improves superelasticity (Saburi *et al.* 1982, Miyazaki & Otsuka 1986, Thoma *et al.* 1995, Otsuka & Ren 1999, Huang & Liu 2001).

From the engineering point of view, mechanical fatigue is defined as the result of a repetitive or fluctuating stress, much lower than that required to cause fracture on a single application of load, which may lead to failure of a device after a period of service. The main characteristic of fatigue failure is that it takes place without any obvious warning, such as permanent deformation or change in the material's structure. Clearly, fatigue resistance is one of the most important aspects to consider when using materials in appliances fitted with rotary parts (Courtney 1990).

Strain levels attained by rotary endodontic instruments during clinical use depend on the root canal geometry and on the applied loads that are concentrated on the region of maximum curvature within the root canal. Of the two parameters used to define canal geometry – radius and angle of curvature – it would seem that radius is the most meaningful, insofar as fatigue resistance of rotary NiTi instruments is concerned (Pruett *et al.* 1997). Furthermore, the importance of geometrical factors in root canal shaping becomes even greater when multiple curvatures are present. The high incidence of secondary curvatures in human mandibular molars (30%) and the fact that they occur predominantly in the apical third of the root canal, at a mean distance of 2.2 mm from the foramen (Cunningham & Senia 1992), demand that NiTi rotary endodontic instruments possess exceptional properties.

Detailed knowledge of how such instruments behave under fatigue is fundamentally important to ensure their safe clinical usage. One relevant question is whether cyclic loading during shaping of curved root canals affects their superelastic properties. The literature has established that, in many cases, the functional properties of NiTi superelastic alloys are affected by cyclic loading (Eggeler *et al.* 2004). This means that NiTi rotary endodontic instruments may have decreased flexibility following repeated use. Considering that the tensile strain amplitude in rotating and bending is the critical component of fatigue, as it promotes crack nucleation and propagation from the surface of the device, attention was directed to tensile cyclic loading. Study of tensile cyclic loading requires homogeneity in the application of stress; ruling out the use of endodontic instruments themselves, as their transverse section at shaft vary continuously along their cutting length. NiTi wires employed in the manufacture of ProFile endodontic instruments are thus the appropriate material to evaluate the influence of cyclic tensile loading on these instruments.

The purpose of this study was to assess the influence of cyclic loading on the mechanical behaviour of NiTi wires employed in the manufacture of ProFile (Dentsply Maillefer, Ballaigues, Switzerland) rotary endodontic instruments, in order to verify the hypothesis that the flexibility of NiTi rotary endodontic instruments decreases through repeated use. The physical and mechanical properties of the wires were also analysed and compared with those of size 30, .06 taper ProFile instruments. In addition, fractured surfaces were observed by scanning electron microscopy (SEM), as the presence of fatigue striations on the fracture surface of previously cycled specimens would indicate that cyclic loading played an important role in the fracture process (Dieter 1986, Courtney 1990).

Materials and methods

NiTi wires were provided by Dentsply Maillefer and were taken from the production line of .06 ProFile rotary endodontic instruments just before the final machining step. Although the maximum diameter, D_{16}, of the size 30, .06 taper instruments is nominally equal to 1.26 mm, the ProFile instruments with .06 taper are machined from a 1.2 mm wire, so that their actual maximum diameter is equal to this value. New size 30, .06 taper ProFile instruments were employed to compare their chemical and physical properties with the NiTi wires.

Specimens cut from the wires in the as-received condition and from new size 30, .06 taper instruments were analysed by X-ray energy-dispersive spectroscopy (EDX) (Noran TN-M3055; Middleton, WI, USA), to determine, semi-quantitatively, their Ni and Ti contents. Ten small areas were analysed in three samples of the wires and in three samples of the instruments. X-ray diffraction (XRD) (Philips-PANalytical PW1710; Almelo, The Netherlands) was employed to identify the phases present in the wires and instruments. Ten 12-mm-long segments of each material were glued side by side forming a specimen 12 × 12 mm in area and analysed, using Cu-Kα radiation. Differential scanning calorimetry (DSC) (Shimadzu DSC 60; Kyoto, Japan), was employed to measure the transformation temperatures. Three tests were performed with different samples of each material. Each test consisted of heating the sample to 80 °C and then cooling to −60 °C, at a heating and cooling rate of 10 °C min^{-1}. Transformation temperatures were determined as the beginning and the end of exothermic and endothermic peaks on the DSC curves.

Specimens of NiTi wires with a 1.2-mm diameter and 80-mm length were tensile tested until rupture in a universal testing machine (Instron 5581; Canton, MA, USA). A slow cross-head speed of 1.5 mm min^{-1}, corresponding to a strain rate of 1.0×10^{-3} s^{-1}, was employed, with an extensometer of 25 mm of gauge length, in order to determine the parameters describing the mechanical behaviour of the wires. These parameters are the transformation stress, $\sigma_{A\text{-}M}$, which is the value of the stress corresponding to the end of the elastic portion of the stress–strain curve in a superelastic material and indicates that austenite begins to transform to martensite; the stress at maximum load, generally called the ultimate tensile strength, σ_{UTS}, which is the maximum stress the specimen can withstand before rupture; and the plastic strain at breakage, called the total elongation, e_T, which is the total permanent deformation imposed in the test, generally expressed as a percentage of the initial gauge length. Three tests performed at identical conditions were employed to evaluate the average values of these parameters.

Cyclic loading tests were carried out with similar NiTi specimens on the same testing machine, at a strain rate of 1.0×10^{-2} s^{-1}. The specimens were loaded in the superelastic plateau to 4% total elongation and unloaded to zero stress. This value of tensile strain is approximately the mean of the estimated values of the maximum tensile strain amplitude at the surface of ProFile instruments during curved root canal shaping, which varies from 3.3% for a size 20, .04 taper instrument to 5.0% for a size 30, .06 taper instrument, in canals with 5 mm of radius and 45 ° of angle of curvature (Bahia & Buono 2005). This cycle was repeated up to 100 times with each specimen (three of each type) and then interrupted. All the stress–strain curves were recorded. The specimens were tensile-tested to failure, at a strain rate of 1.0×10^{-3} s^{-1}. As before, transformation stress, stress at maximum load and plastic strain at breakage of the cycled wires were evaluated as the average of three tests.

The fracture surfaces of the wire specimens tensile-tested to failure in the as-received condition and after 100 load–unload cycles were observed by SEM in a Jeol JSM 6360 (Tokyo, Japan).

Results

The EDX semi-quantitative chemical analysis of the NiTi wires and the ProFile instruments showed that, on the average (10 areas on each specimen, SD lower than

Table 1 Average values of the transformation temperatures (±SD) determined by differential scanning calorimetry

Material	Transformation temperatures (°C)			
	Ms	Mf	As	Af
ProFile 30/.06	18.2 ± 0.4	−2.3 ± 3.6	3.4 ± 4.7	22.9 ± 1.0
NiTi wires	17.5 ± 1.3	−5.0 ± 1.2	1.1 ± 1.1	21.8 ± 3.6

Ms, martensite start temperature; Mf, martensite finish; As, austenite start temperature; Af, austenite finish. NiTi, nickel-titanium.

Table 2 Mean values (±SD) of the transformation stress ($\sigma_{A\text{-}M}$), stress at maximum load (ultimate tensile strength, σ_{UTS}) and plastic strain at breakage (total elongation, e_T)

Property	Condition	
	As received	Cycled 100 times
$\sigma_{A\text{-}M}$ (MPa)	550 ± 7.5	404 ± 21.3
σ_{UTS} (MPa)	1404 ± 7.0	1403 ± 18.1
e_T (%)	11.2 ± 0.9	12.4 ± 0.3

0.5%), both materials had the same composition, namely 51 at.% Ni-49 at.%Ti (56 wt% Ni-44 wt%Ti), which is the chemical composition reported by the manufacturer (Dentsply Maillefer). XRD analyses of wire and instrument samples also gave similar results, indicating that, at room temperature, they both had the β-phase, with the ordered body-centred cubic structure named B2, as the main constituent. The average transformation temperatures (Ms, Mf, As, Af), determined by DSC in specimens of the NiTi wires and of size 30, .06 taper ProFile instruments, are shown in Table 1.

The average stress–strain curves obtained from three specimens of the NiTi wires in the as-received condition and from three specimens previously submitted to 100 load–unload cycles are shown in Fig. 1. The mean values and SDs of the transformation stress, ultimate tensile strength and plastic strain at breakage, determined for the as-received and cycled wires, are shown in Table 2. The stress at maximum load and the plastic strain at breakage are practically the same (the change in e_T is of the order of the SD). In fact, the main effect of cyclic loading was decreasing $\sigma_{A\text{-}M}$ by about 26.5% and increasing the strain at the superelastic plateau by approximately 25%. Typical stress–strain curves obtained in cyclic loading tests are shown in Fig. 2 for the first cycle and for cycles number 12, 25, 50, 75 and 100, for illustration. The tendency towards stabilization of the mechanical behaviour of the NiTi wires, after cycle number 12, is clearly demonstrated in this figure.

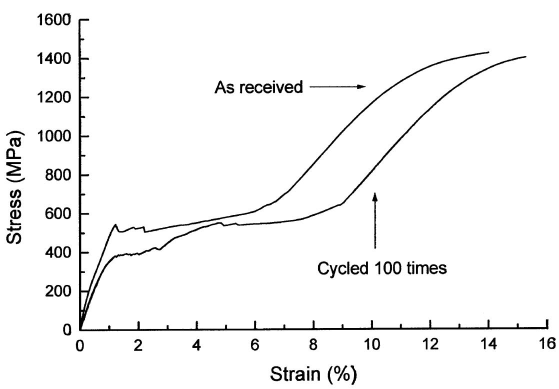

Figure 1 Average stress–strain curves of the nickel-titanium wires in the as received condition and after 100 load–unload cycles.

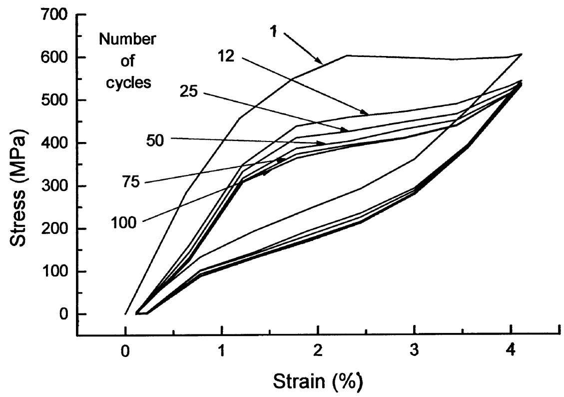

Figure 2 Typical stress–strain curves obtained in cyclic loading tests.

The fracture surfaces of as-received and cycled wires tensile-tested to failure are shown in Fig. 3. Both surfaces display the characteristic cup-and-cone fracture of ductile metals, with a peripheral shear area surrounding the fibrous central region. Details of the fibrous central region of the fracture surfaces can be seen in Fig. 4, revealing the presence of dimples and slip lines, as expected for this type of failure (Dieter 1986, Courtney 1990). The shear area on the fracture surface of the cycled specimen is shown in higher magnification in Fig. 5, where the presence of fatigue striations and numerous secondary cracks can be observed. Similar features could not be detected on the shear area of wire specimens tensile-tested to failure in the as-received condition, indicating that these specimens failed by a different mechanism.

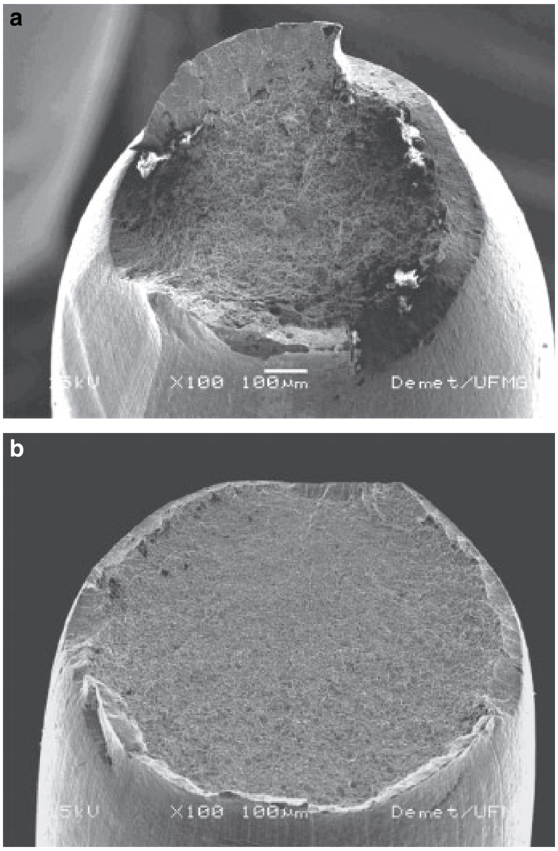

Figure 3 Fracture surfaces of as received (a) and cycled (b) wires tensile-tested to failure. Secondary electron images by scanning electron microscopy. Original magnification: 100×.

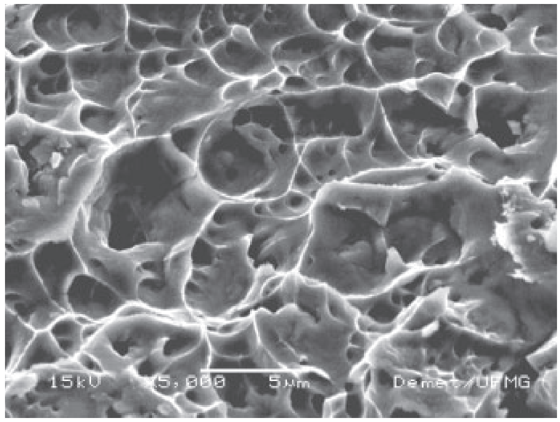

Figure 4 Fibrous central region of the fracture surfaces. Secondary electron images by scanning electron microscopy. Original magnification: 5000×.

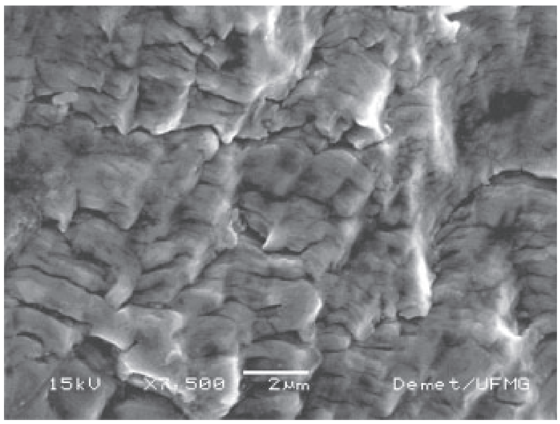

Figure 5 Shear area on the fracture surface of a cycled specimen. Secondary electron images by scanning electron microscopy. Original magnification: 7500×.

Discussion

The fact that the presence of Ti_3Ni_4 precipitates could not be detected in the specimens investigated by XRD in this work is an indication that their amount is lower than the resolution of the technique employed, approximately 3% in volume. For such small amounts of Ti_3Ni_4 precipitates, it is reasonable to expect that the formation of the R-phase, whose nucleation is strongly dependent on the stress fields around these coherent precipitates (Allafi *et al.* 2002), would not occur in the wires studied. These observations are in agreement with that found in the DSC analysis (Table 1): only the transformation of austenite to B19′ martensite on cooling, and the reverse transformation of B19′ martensite to austenite upon heating, were detected.

Similar DSC results were obtained by Kuhn & Jordan (2002) in size 20, .04 taper ProFile instruments. However, the temperatures reported by those authors for the martensitic and the reverse transformations, respectively 35°C and 39°C, are higher than those shown in Table 1 and are not compatible with the occurrence of the superelastic effect in the conditions appropriate for the clinical use of the instruments. These values of transformation temperatures indicate that the superelastic behaviour, and thus the flexibility which is the basis of the rotary technique with NiTi instruments and takes place above Af, would not occur in clinical practice, that is, between room temperature and body temperature, 37 °C. In general, results of DSC analyses in NiTi endodontic instruments are contradictory. For instance, Brantley *et al.* (2002) found, for unused ProFile instruments, values of the As temperature as low as −33 °C, which certainly should not be expected for NiTi superelastic alloys (Otsuka & Wayman 1998, Liu *et al.* 1999).

The tensile stress–strain curves shown in Fig. 1 disclose important characteristics of the behaviour of NiTi superelastic alloys. The stress peak at the beginning of the superelastic plateau corresponds to the nucleation of martensite variants in austenite, while the subsequent decrease in stress occurs because the propagation of these convenient orientated martensite variants requires lower stresses (Krishnan *et al.* 1974, Shaw & Kyriakides 1995, Huang & Liu 2001).

Comparison of the values of the parameters describing the mechanical behaviour of the NiTi wires (Table 2) indicates that this behaviour is only slightly modified after 100 load–unload cycles up to 4% tensile strain. The use of a higher strain rate in the cyclic tests aimed to decrease the total test time and should not influence the results, because it is also a slow strain rate, corresponding to a cross-head speed of 15 mm min^{-1}. Tolomeo *et al.* (2000), comparing the mechanical properties of superelastic NiTi stents under monotonic and cyclic loading, found that appreciable stress changes because of cyclic loading took place mainly at the superelastic plateau, in agreement with the results found in the present work. The stress–strain curves shown in Fig. 2 confirm the observation that the transformation stress decreases as the number of load–unload cycles increases. They also show that the stress for the reverse transformation upon unloading and the stress hysteresis also decrease, while the nonrecoverable strain increases from about 0.1 to 0.25% after 100 load–unload cycles. It is also important to observe that the changes in the tensile behaviour of the NiTi wires during cyclic deformation take place mainly in the initial cycles, tending to stabilize as the number of cycles increases. The observed changes are usually associated with the internal defects generated when NiTi alloys are submitted to cyclic deformation in the superelastic regime (Tobushi *et al.* 1996, McKelvey & Ritchie 1999). The nonrecoverable strain is attributed to the generation of dislocations and the presence of nontransformed martensite variants near grain boundaries after unloading. The internal stresses associated with these defects contribute to the nucleation of stress-induced martensite during subsequent loading, thus decreasing the required transformation stress. The saturation in the amount and distribution of the internal defects after the initial load–unload cycles is responsible for the observed stabilization effect. These observations have important consequences for the clinical use of ProFile instruments: the increase in the nonrecoverable strain is small and should not affect the flexibility of the instrument. On the contrary, the increase in the transformation strain means that the instruments can flex more in the superelastic plateau, before suffering permanent deformation. The decrease in the transformation stress causes reduction of the restoring force, which may be beneficial to canal shaping.

The presence of fatigue striations in the shear area of the fracture surfaces of the cycled wires (Fig. 5) indicates that the failure of these specimens in the tensile tests involved pre-existing fatigue cracks, developed during load–unload cycling. Fracture surfaces of the as-received wires tensile tested to failure did not show such characteristics, meaning that they failed by tensile overload alone (Dieter 1986, Courtney 1990). This observation has also important consequences when the results presented in this work are considered in a more practical basis, i.e. that of the clinical use of ProFile endodontic instruments. A necessary condition for the safe use of this type of instrument is that the tensile strain component developed in the instrument's surface when it rotates inside a curved root canal remains in the superelastic plateau, otherwise permanent deformation takes place. What was shown here is that the superelastic behaviour of the NiTi wires from which the ProFile instruments are made is not significantly affected by cyclic loading in tension, up to 100 load–unload cycles, within this strain region. Nevertheless, the accumulation of the internal defects generated during cyclic loading, although not producing unfavourable changes in the superelastic properties, will eventually cause fatigue failure of the instruments. In fact, they will also affect their torsional properties, decreasing the maximum torque the instruments can withstand in torsion (Yared & Kulkarni 2003, Bahia 2004). Furthermore, clinical-related factors such as varying apically directed force, speed of insertion and time required for shaping can differ greatly, consequently affecting the risk of instrument failure.

Conclusions

The mechanical behaviour of NiTi wires employed in the manufacture of ProFile endodontic instruments is only slightly modified by cyclic tensile loading in the superelastic plateau, meaning that the ProFile instruments maintain their major superelastic characteristics after use for cleaning and shaping curved root canals.

However, the accumulation of internal defects associated with cyclic deformation indicates that used instruments may fail because of fatigue or torsional overloading.

Acknowledgements

This work was partially supported by Fundação de Amparo à Pesquisa do Estado de Minas Gerais – FAPEMIG, Belo Horizonte, MG, Brazil, and Conselho Nacional de Desenvolvimento Científico e Tecnológico – CNPq, Brasília, DF, Brazil.

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