JOURNAL OF ENDODONTICS

Printed in U.S.A.
VOL. 28, NO. 10, OCTOBER 2002

Fatigue and Mechanical Properties of Nickel-Titanium Endodontic Instruments

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Shape memory alloys are increasingly used in superelastic conditions under complex cyclic deformation situations. In these applications, it is very difficult to predict the service life based on the theoretical law. In the present work, fatigue properties of NiTi engine-driven rotary files have been characterized by using differential scanning calorimetry (DSC) and mechanical testing (bending). The DSC technique was used to measure precise transformation. The degree of deformation by bending was studied with combined DSC and mechanical property measurements. In these cold-worked files, the high dislocation density influences the reorientation processes and the crack growth. Some thermal treatments are involved in promoting some changes in the mechanical properties and transformation characteristics. Annealing around 400°C shows good results; the recovery allows a compromise between an adequate density for the R-Phase germination and a low density to limit the brittleness of these instruments. In clinical usage, it is important to consider different canal shapes. It could be proposed that only few cycles of use is safe for very curved canals but to follow the manufacturer's advise for straight canals.

In endodontic treatments, the risk with traditional files (stainless steel) is plastic deformation and fracture. Consequently, nickel-titanium (NiTi) instruments with pseudo-elasticity properties (shape memory effect and superelasticity) have been introduced to avoid or to limit the failure risk. However, cyclic deformation during endodontic treatment changes the mechanical behavior of NiTi alloys and finally leads to fatigue failure.

The superelasticity (SE) nature of NiTi has been attributed to a reversible austenite to martensite transformation. It is believed austenite is transformed to martensite during loading and reverts back to austenite when unloaded. The transformation is reversible during clinical use, because SE alloys have a transition temperature range (TTR) lower than mouth temperature. The TTR of NiTi is effected by the chemical composition, method of fabrication, and heat treatment of the alloy (1). Sometimes, the direct transformation from austenitic to martensitic NiTi includes an intermediate structure, called R-phase. It is important to have knowledge of the relationships between Austenite, R-phase, and Martensite transformation sequences on cooling and heating. On cooling, we can observe: Austenite → R-phase → Martensite (direct transformation) and on heating: Martensite → R-phase → Austenite (reverse transformation). However, due to the large differences between the hysteresis of the martensite and the R-phase transformations, in some cases, the transformation sequence on heating is Martensite → Austenite directly. The transformation A → R-phase shows the same properties (superelasticity and shape memory effect) because of the quasi-martensitic nature of this transformation. Young's Modulus of the R-phase is lower than that of Martensite, and thus, an instrument with the R-phase transformation would be more flexible.

The mechanical properties and various phase transformation temperatures of NiTi shape memory alloy are known to be very dependent on thermo-mechanical processing. To use these alloys in various applications, proper control and understanding of the effects of thermo-mechanical processing parameters are very essential. We used some thermal treatments that are involved in promoting some changes in the mechanical properties and transformation characteristics. These properties can be modified by high dislocation density and/or fine dispersion of particles. A variety of irreversible phenomena associated with dislocations, precipitates, and residual stresses from previous cold work and/or thermal histories complicates the exploitation of superelastic (SE) alloys.

The aim of this work is to show fatigue characteristics of superelastic NiTi, and subsequently, the effect of the process history on fracture life. This work is the continuation of our results based on microstructural investigations (scanning electron microscopy, X-rays diffraction, and microhardness) of nickel-titanium instruments (2). We investigated mechanical properties with emphasis on flexibility of endodontic instruments by the use of bending tests. This study will then be discussed using differential scanning calorimetry (DSC) results. DSC allows the identification of crystallographic phases at various temperatures (3).

MATERIALS AND METHODS

Materials

The engine-driven, rotary instruments that were studied are produced by Maillefer (ProFile, Ballaigues, Switzerland) and by

TABLE 1. NiTi specimens

Conicity-Diameter	Hero	ProFile
0.02/30	◆	
0.04/20		◆ ✲ ▲
0.04/30	◆	◆
0.06/20		◆ ☆
0.06/30	◆	◆

◆ = new instrument, active part; ✲ = new instrument, inactive part; ☆ = used instrument, active part; ▲ = instrument after thermal treatments, active part.

Micro-Mega (Hero, Besançon, France) in many geometrical shapes (Table 1). The studied files have a 25-mm length, a taper ranging between 0.04 and 0.06 mm per mm length, and sizes 20 to 40, representing the diameter of the tip base of the file, given in hundredth of millimeter.

Specimens were cut to separate working or active part of the file from the inactive part using a low-speed diamond saw. Several samples were chosen: new instruments and instruments that have been used in clinical conditions (12 or 18 root canals and approximately 5 or 6 sterilizations).

Thermal Treatments

Different thermal treatments were investigated. The heat treatments consisted of anneals at 350°C, 400°C, 450°C, 510°C, 600°C, and 700°C in salt baths for 10 min and at 600°C and 700°C for 15 min with the same process and subsequent water quench in all cases.

Machining process promotes a high density of defects; the alloy is work-hardened. We used some thermal treatments, which are involved in promoting changes in the mechanical properties and transformation characteristics. The influence of different heat treatments on NiTi alloys was investigated by DSC measurements and bending tests.

Methodologies

DIFFERENTIAL SCANNING CALORIMETRY

DSC testing is one of the many test methods used to measure transformation temperatures of NiTi alloys. DSC testing is a thermal method that measures the change in heat flow, which is associated with the martensitic and austenitic phase transformations through a controlled cooling/heating cycle. In the DSC procedure, the differential heat flow required to heat or cool the experimental and reference samples at the same scanning rate is recorded as a function of temperature to yield the spectrum or thermogram. The start and finish temperatures of each phase transformation were determined from tangent lines where the DSC curve deviates from the adjacent baselines.

The transformation temperatures were determined by DSC (Mettler 30/TA 4000). The file specimens were carefully cut with an oil-cooled, diamond-embedded saw. Considerable care was taken in cutting the samples so that minimal heat and stress would be generated. Straight segments of 5-mm length were cut from each file. Specimens were placed in aluminum pans with a nitrogen gas flow environment to prevent condensation of water vapor on the NiTi specimens. Another empty aluminum pan served as an inert reference. The weight of the samples for DSC measurements was 18 mg; heating and cooling rates were 5°C/min. The thermograms were carried out in temperature intervals of +60°C and −120°C. During measurements, the samples were quickly heated to 60°C and then cooled to approximately −120°C at a constant cooling rate (5°C/min). When the low temperature was reached, the specimen was heated again to 60°C at the same rate.

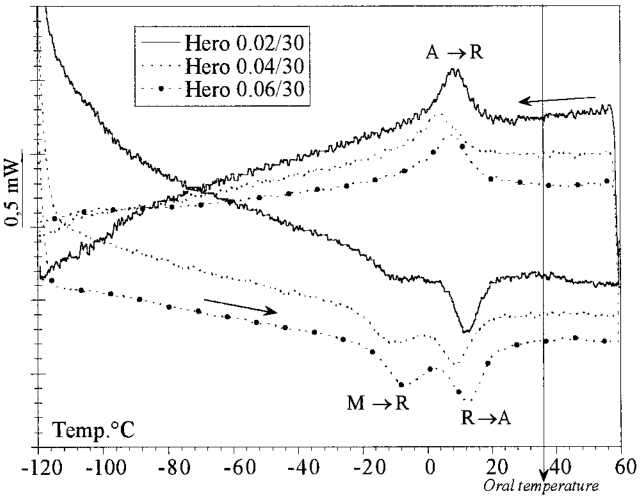

FIG 1. DSC thermograms obtained for different conicity.

Bending Tests

To perform bending tests, we used a bending testing machine. All the instruments were loaded with the same deformation, and the forces corresponding were calculated by the cell (100 N). The loading and the unloading were performed in the same conditions.

We tried with our machine to reproduce the bending of files that occurs in clinical situations. New instruments, instruments used in the clinic, and instruments that have been heat-treated were included in this mechanical test. We obtained information about the elastic behavior (flexibility) of files and about heat treatments and clinical use. The results are discussed only in a qualitative analysis and not a quantitative analysis because of the shape of the instruments (range and machining design), which prevents any calculation.

RESULTS

DSC

The transformation process can easily be recorded by measuring the transformation latent heat released/absorbed to/from the surroundings. Figure 1 shows the DSC curves for different conicities of Hero instruments. DSC curves show one-step distinct transformation during cooling. The transformation product of the first exothermic peak at higher temperature corresponding to the transformation from austenite (A) to R-phase (R), whereas that of the second exothermic peak at lower temperature is difficult to identify and is dissimulated in the baseline of the thermogram. Two-step endothermic transformation occurs during heating (M → R and R → A).

The peaks are better and better defined with increasing conicity. The ProFile samples show the same characteristics, but all peaks are less defined. In the oral environment, the specimen are completely austenitic or a mixture of austenite and R-phase.

TABLE 2. Transformation temperatures

	Direct transformation TTR (°C)	Reverse transformation (°C)
Hero	20	23
ProFile	35	39

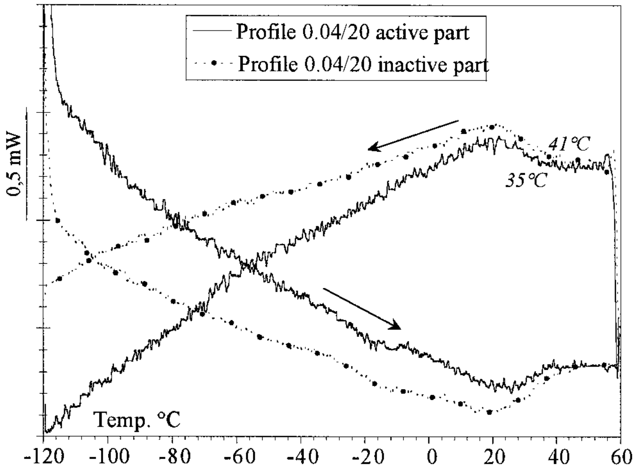

FIG 2. DSC curves obtained from different part of the instrument.

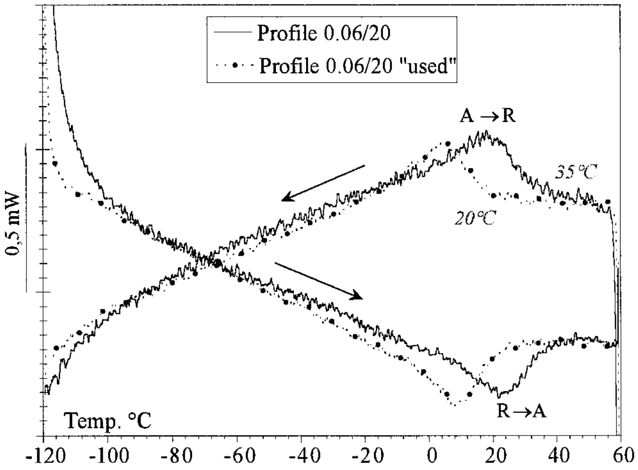

FIG 3. DSC thermograms showing new ProFiles and ProFiles that have been used in clinical conditions.

Table 2 summarizes the main transformation temperatures for each file.

DSC cooling and heating curves in Fig. 2 show peaks associated with the A ↔ R transformation but poorly resolved peaks; we can observe two different TTR for the active part (35°C) and inactive part (41°C).

Figure 3 shows the DSC curves for new ProFiles and ProFiles that have been used in clinical conditions (12 root canals and approximately 10 sterilizations). In the case of the new files, one peak is obtained on cooling and on heating (A ↔ R). However, when the file is used, the same peaks are observed but the TTR shifts to a lower temperature: approximately 15°C lower. The R ↔ M transformation does not appear.

After heat treatments, the samples show two different types of transformation courses during DSC measurements. The peaks intensity and position change with varying annealing conditions.

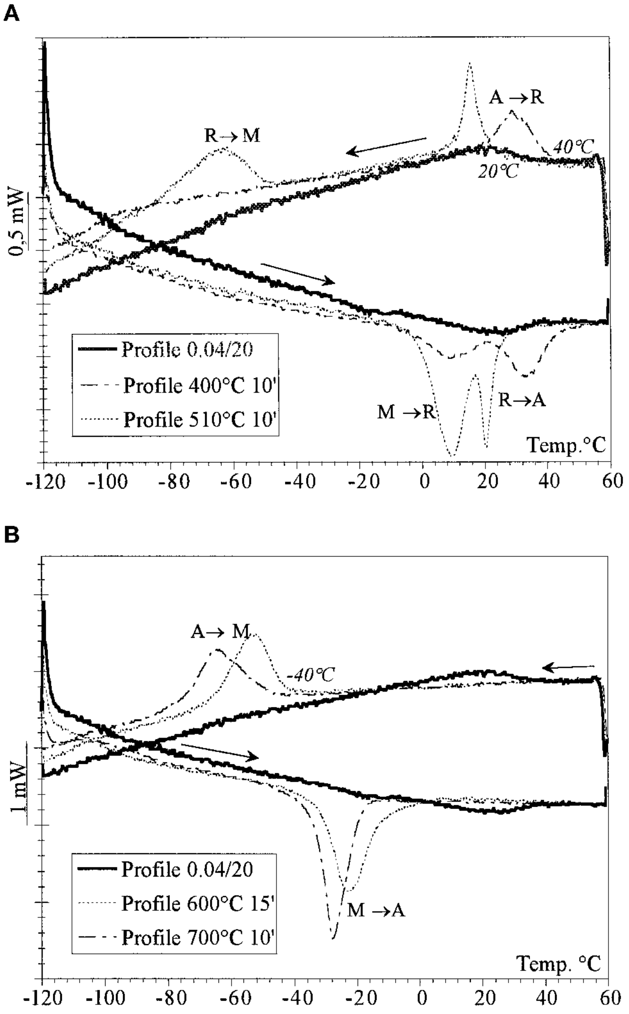

FIG 4. (*A*) DSC curves after heat treatments below 600°C. (*B*) DSC curves after heat treatments above 600°C.

After heat treatments below 510°C (Fig. 4A), two peaks are found during cooling process (A → R, R → M) and two peaks during heating (M → R, R → A). Above 510°C, thermal treatments yield a transformation behavior with one peak during cooling and during heating (Fig. 4B). When the annealing temperature is above the recrystallization temperature (600°C), only one exothermic peak can be found during cooling (A → M). We have the direct and reverse A ↔ M transformations; the R-phase transformation does not exist. This thermal treatment shifts the martensite transformation to lower temperatures. For these different thermal treatments, DSC thermograms show TTR evolution (Table 3).

Bending Tests

At first, and until 3 mm of strain, only the tip of the instrument is bent. Then, between 3 and 6 mm, the curvature is in the middle of the file. Finally, above 6 mm, the part that has the maximum cross-sectional area near the handle becomes deformed in turn.

As can be seen from the curves, the samples deformed at room temperature recover their original state, indicating that the transformation temperature is close to room temperature. Specimens seem to exhibit significant plateau-stress drop or stress relaxation during superelastic cycling.

TABLE 3. TTR evolution after heat treatments

	Before Heat Treatment	400°C	510°C	600°C	700°C
TTR	35°C	40°C	22°C	−42°C	−46°C

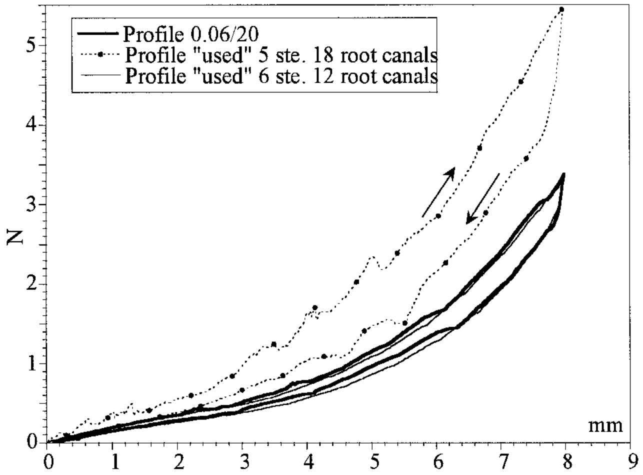

FIG 5. Bending curves showing new ProFiles and ProFiles that have been used in clinical conditions.

We cannot notice any difference between a new instrument and a file that has been used in straight canals (Fig. 5). But when the abruptness of canal curvature increases, the stiffness of the used file increases after each use.

Figure 6 (A and B) demonstrate that the annealing conditions strongly affect the stress-strain behavior. For heat treatments below recrystallization temperature (Fig. 6A), the specimens generally show an increased flexibility. On the other hand, results show that after annealing at a temperature above recrystallization, the stiffness of the instruments increases (Fig. 6B).

DISCUSSION

Results previously presented with XRD and microhardness (2) and our results with DSC show that new specimens are significantly work-hardened. The microhardness measure is twice that of a fully recrystallized sample (4). Moreover, machining marks and cracks on the surface observed by scanning electron microscopy contribute largely to fatigue failure by a crack propagation process. The crack nucleation stage is improved by the high density of surface defects. Manufacture of NiTi alloys by machining into endodontic instruments promotes work hardening and creates surface defects. These first observations (microstructure and surface defects) explain unexpected fractures reported in the literature (5).

A variety of irreversible phenomena associated with dislocations, precipitates, ordering effects, and residual stresses from previous cold work and/or thermal history complicates the use of shape memory alloys. The substantial variance in the thermograms of active and inactive parts results from the manufacturing processes. The active part is more affected by machining compared with the inactive part (Fig. 3). When the deformation temperature (oral temperature) lies near Ms on initial loading, a martensite and subsequently R-phase are stress induced and the latter is stable throughout the rest of the test. Those martensite (or R-phase) variants will be selectively produced, which gives maximum strain

stress reversal probably leads to defect formation. Dislocations present in the matrix influence the mechanical properties; internal stresses are a negative factor to the mobility of martensite interfaces (6). Moreover, DSC thermograms (Fig. 3) and bending curves (Fig. 5) of used instruments (abrupt curvatures) show an increased density of dislocations. Indeed, the shift of the TTR to lower temperature impedes the phase transformation; instruments become stiff. It seems that the abruptness of the curvature of the root canals (stress field) is the essential parameter and that the numbers of root canals treated is less important for the increase of brittleness. Manufacturers advise not to use each file on more than 10 to 12 root canals. The difference between files that have been stressed in curved canals compared with straight canals can be seen in terms of different defect densities created by martensite reorientation. In the curved canals, the stress to induce martensite will be high. The build-up of stress concentrations could be prevented from forming cracks, because martensite will be retained to accommodate this stress. But if the density of defects is high (in case of work hardening), the reorientation of variants in the stress field or its reversion and reformation is not possible and cracks appear and grow.

In Figs. 3 and 5, specimen behavior is probably a consequence of dislocations and lattice defects generated by high stress levels. When lattice defects are created, they will restrict the easy movement of martensite, i.e. they decrease the mobility of martensite interfaces.

It was found that martensite transformation propagated in steel retarded crack growth, and it was proposed that this could result from internal compressive stresses induced by a positive volume change near the crack tip. However, in NiTi the volume change is small and negative and thus causes a negligible effect of stress-induced martensite near a growing crack (7).

According to Yang (8), the fracture life apparently obeys the following relationship if high strain (curved canal) and low strain are considered separately according to different deformation mechanisms:

$$\Delta\epsilon \cdot N^{\beta} = C$$

where C and β are constants, $\Delta\varepsilon$ is the applied strain amplitude, and N is the number of cycles to fractures. For an optimization of fatigue resistance in the specific range of reversible deformation, it is necessary to pay attention to the shape of canals. The fatigue behavior of superelastic files is highly dependent on strain level and heat treatment conditions.

Heat treatments are known to influence mechanical properties and various phase transformation temperatures of NiTi shape memory alloys. Two annealing temperature ranges can be distinguished. In the first case, annealing temperatures approximately 600°C (recovery) show two-step transformations (A ↔ R ↔ M); in the second case, when annealing temperatures are above 600°C (recrystallization), we can observe a direct martensitic transformation. The annihilation of dislocations by recovery and recrystallization, or the beginning of dissolution of precipitates, are of special importance both for the structural properties and the functional properties (e.g. transformation temperatures). It can be supposed

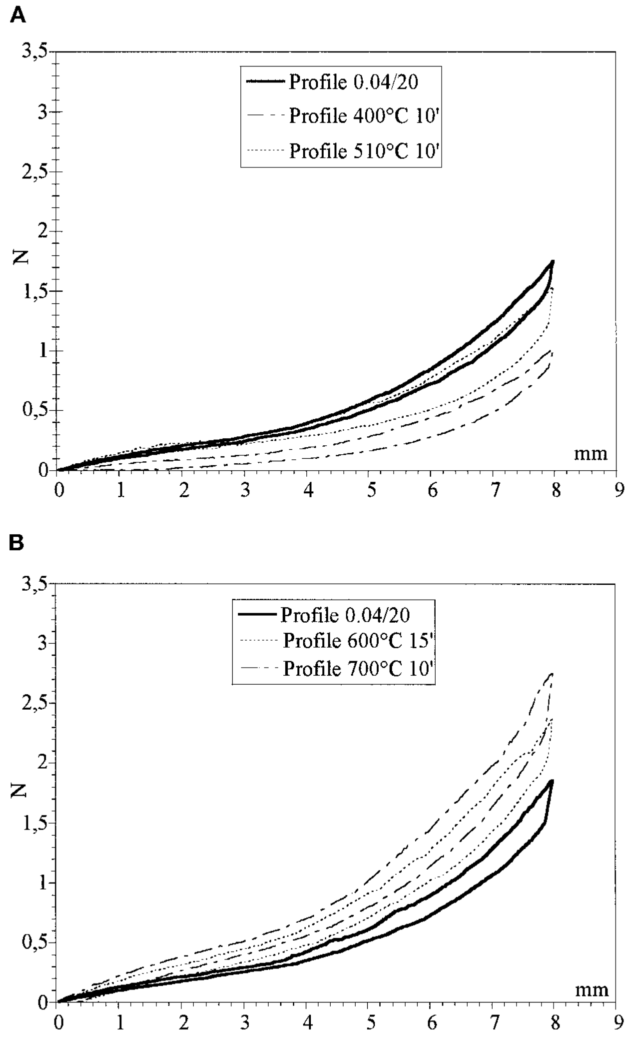

FIG 6. (*A*) Bending curves for various annealing conditions. For heat treatments below 600°C, the specimens show an increased flexibility. (*B*) Bending curves for various annealing conditions. For heat treatments above 600°C, the stiffness of the instruments increases.

differently depending on whether there are many dislocations during annealing. Due to the thermal activation, the voids will move into the fields of compressive stress (inside the dislocations) and reduce the number of nucleation sites in a microstructure with higher dislocation density. With beginning recrystallization and dissolution of the particles after further annealing, no more R-phase transformation can be detected. With the beginning recrystallization (600°C and 700°C) (Figs. 4B and 6B), R-phase transformation cannot be detected. It can be presumed that the decreasing dislocation density and any precipitation stress fields are not able to initiate the R-phase transformation. The dissolution of Ni-rich precipitates increases Ni-content in the matrix and shifts the TTR to a lower temperature. Thus, the stiffness is much more important. For clinical applications, these heat treatments are not required.

The shape memory alloy files are expected to show unique fatigue characteristics different from the usual materials because of their deformation behavior associated with the martensitic transformation. In fact, fatigue crack propagation rate and fatigue life strongly depend on test temperature (9); such strong temperature dependence is unique and cannot be found in the standard data of ordinary materials.

Various degradation modes occur, including a shift in transformation temperature, a reduction in the available strain, and of course, fracture. With regard to the fatigue behavior in the superelastic temperature range, fracture is a critical failure mode for many applications.

In these cold-worked files, the high dislocation density influences the reorientation processes and the crack growth. The instruments become brittle. As to superelasticity, with cycling reorientation of the martensite under stress leads to gradual defect accumulation, and it might be expected that these dislocations are generated at the interface between different martensite colonies. In clinical conditions, the curvature of canals distorts the endodontic instruments; cyclic fatigue is caused by repeated tensile-compressive stress. The maximum of this stress is in the surface of the curve. Crack nucleation and propagation stages appear mostly on the half-part of the instrument, which is in tension (outside of the curve).

Some suggestions could be proposed to improve the lifetime of endodontic files; these include applying thermal treatments at approximately 400°C (recovery) before machining to decrease the work-hardening of the alloy, choosing machining conditions adapted to this NiTi shape memory alloy, and electropolishing by the manufacturer to reduce the machining damage on the file surface. For an optimization of the fatigue resistance in the specific range of reversible deformation, it is necessary to pay attention to shape of the canal. Only a few cycles of use for very curved canals may be best, and following the manufacturer's advice may be best for straight canals. Therefore, it is very important to understand the fatigue characteristics of NiTi alloys to use these functions in various types of applications.

The authors are grateful to Dr. France Dalle for reviewing the manuscript.

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