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Surface characterization of retrieved NiTi orthodontic archwires

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SUMMARY The structure and morphological condition of retrieved NiTi orthodontic archwires was evaluated and any possible alterations in the surface composition of the alloy following 1–6 months *in vivo* were characterized. NiTi wires (GAC, German Orthodontics, ORMCO) of various cross-sections were collected through a retrieval protocol and were subjected to multi-technique characterization.

Optical microscopy revealed islands of amorphous precipitants and accumulated microcrystalline particles. Micro MIR-FTIR investigation of the retrieved samples demonstrated the presence of a proteinaceous biofilm, the organic constituents of which were mainly amide, alcohol, and carbonate. Scanning electron microscope and X-ray microanalysis showed that the elemental species precipitated on the material surface were Na, K, Cl, Ca, and P, forming NaCl, KCl, and Ca-P precipitates. Increased intra-oral exposure was consistently associated with the presence of a mature film, while evidence of alloy delamination, pitting, and crevice corrosion, as well as a notable reduction in the alloy grain size was observed.

Intra-oral exposure of NiTi wires alters the topography and structure of the alloy surface through surface attack in the form of pitting or crevice corrosion or formation of integuments. Further *in vivo* research is required to resolve the implications of the described ageing pattern in the corrosion resistance of the alloy, the potential for nickel leaching, as well as bracket-archwire friction variants.

Introduction

The use of nickel-titanium archwires as an initial wire in the levelling and alignment stages of treatment has expanded significantly since their introduction in the early 1970s (Andreasen and Brady, 1972). The large springback, controlled stiffness (Andreasen *et al.*, 1985), and high stored energy possessed by these wires (Burstone and Goldberg, 1983), have facilitated imaginative approaches in mechanotherapy, such as early full bracket slot engagement, leading to the introduction of the differential modulus concept (Burstone, 1981). In addition, the low load-deflection ratio over a wide range of deformation for this alloy (Kapilla and Sachdeva, 1989), has contributed to the establishment of long intervals between appointments, reducing the required screening visits, and thereby facilitating long active periods which may exceed 6 months.

Although much research has focused on the detailed study of the mechanical properties (Khier *et al.*, 1991) and phase transformation changes of these wires (Bradley *et al.*, 1996), there is a scarcity of information on the effect of the intra-oral environment on the surface structural and compositional alterations. The lack of relevant evidence may derive from the noted inability of *in vitro* research to simulate *in vivo* conditions reliably because of the multiplicity and potency of factors present in the oral cavity, such as extreme pH and temperature variations, and the complex oral flora and its byproducts.

In the case of *in vivo* aged orthodontic wires, the foregoing considerations pertain to the biocompatibility of the archwire, the corrosion resistance of the alloy, and the implication of wire properties in the clinical performance of these wires. The proposed sequelae involves potential hypersensitivity reactions and biocompatibility concerns (Jones *et al.*, 1986) assigned to dissolution of nickel documented to occur *in vivo* (Oshida *et al.*, 1992), corrosion fatigue or stress corrosion cracking attributed to nucleation of pitting or crevice corrosion sites acting as stress risers or crack starters (Edie *et al.*, 1981), and tribological considerations involving the presumably increased friction noted during sliding of these wires on bracket slots due to the extended roughening of the surface.

The majority of the few published reports analysing *in vivo* aged wires, has focused on the study of corrosion resistance (Oshida *et al.*, 1992) and surface morphology through microscopic examination of the retrieved archwire surfaces (Mohlin *et al.*, 1991; Grímsdóttir and Henstenpettersen, 1997). However, no information on the material surface composition relative to topographical features has been reported.

The hypothesis tested in this investigation was that the complex conditions present in the oral cavity, including plaque accumulation and organization of biofilm on the exposed surfaces, substantially alter the surface properties and structural conformation of the archwires. Therefore, the structure and morphology of retrieved wires were evaluated, and possible alterations in the surface composition of nickel-titanium archwires following 1–6 months *in vivo* were characterized.

Materials and methods

The NiTi wires analysed in the study (German Orthodontics, California, USA; Bioforce GAC International, Central Islip, New York, USA; and Neosentalloy, ORMCO, Glendora, California, USA) were retrieved during the regular treatment visits of orthodontic patients to the Graduate Orthodontic Clinic of the School of Dentistry, at the Aristotle University of Thessaloniki, Greece. The patients were selected randomly from a pool of participants established with the following criteria: similar age range, no medication or other intra-orally administered substances, and similarity in gross malocclusion parameters, i.e. crowding less than 5 mm and no significant rotations. Each graduate student was provided with self-closed sterilizing plastic bags, and was instructed to monitor all NiTi archwire insertion and retrieval appointments by means of a retrieval protocol consisting of the following variables: (a) name of patient and graduate student; (b) date of archwire placement; (c) archwire brand cross-section and dental arch of insertion; (d) method of ligation, i.e. stainless steel or elastic; and (e) date of archwire removal. All brackets used at the clinic were of 0.018-inch slot size.

Archwire retrieval procedure yielded approximately 60 retrieved archwires mostly of 0.016-inch and 0.016×0.022-inch cross-sections placed intra-orally for a period of 1–6 months. The collected archwires were rinsed with double distilled water to detach any loose bound precipitations, and specimens of 10 mm length were prepared from molar and incisor regions of each archwire. An identical specimen construction procedure was followed for unused archwires of manufacturer and size, matching each one of the retrieved specimens. All specimens were then subjected sequentially to multi-technique characterization.

The morphological appearance of the surfaces was investigated by reflected light microscopy (Microphot Nikon, Kogaku, Tokyo, Japan) operating in brightfield and polarized light modes.

The molecular composition of the integuments formed intra-orally on specimen surfaces was studied by micro-multiple internal reflectance Fourier transform infrared spectroscopy (micro-MIR FTIR). Spectra acquisition was performed on an FTIR spectrometer (PE 1760 X, Perkin Elmer Corp., Norwalk, CT, USA) equipped with a micro-MIR attachment operating under the following conditions: 4000–400 cm^{-1} range, 4 cm^{-1} resolution, 50 scans co-addition, KRS-5 mini-crystal of 45 degrees edge, and 14 internal reflections.

Scanning electron microscopy and wavelength dispersive electron probe micro-analysis

(SEM-WDS) were used to assess the elemental composition of the features related to the micro-morphological changes induced on wire surfaces after intra-oral exposure. For this purpose, specimens were vacuum-coated with a thin layer of conductive carbon and examined under an electron probe micro-analyser (JXA 733 Superprobe, Jeol Ltd., Tokyo, Japan). Secondary electron images (SEI) and back-scattered electron images for topography (BEI-TOPO) and composition (BEI-COMPO) were recorded at 20 KV accelerating voltage, 8 nA probe current and 5 nA specimen current. Elemental analysis was performed with two spectrometers equipped with PET, TAP, LIF, and STE crystals utilizing area scan mode analysis.

Representative specimens were subjected to metallographic analysis after polishing and etching to evaluate the morphological and structural changes of wire surfaces relative to bulk. The specimens were embedded in epoxy resin and polished using 320–4000 grit size SiC papers and 3-μm diamond paste on a polishing unit (DAP-V, Struers, Copenhagen, Denmark). Some specimens were further etched with an etching solution of concentrated hydrofluoric, nitric, and acetic acids at 1:1:1 volume ratio to reveal the martensitic structure of the alloy (Montero-Ocampo *et al*., 1996). All specimens were then studied under reflected light microscopy.

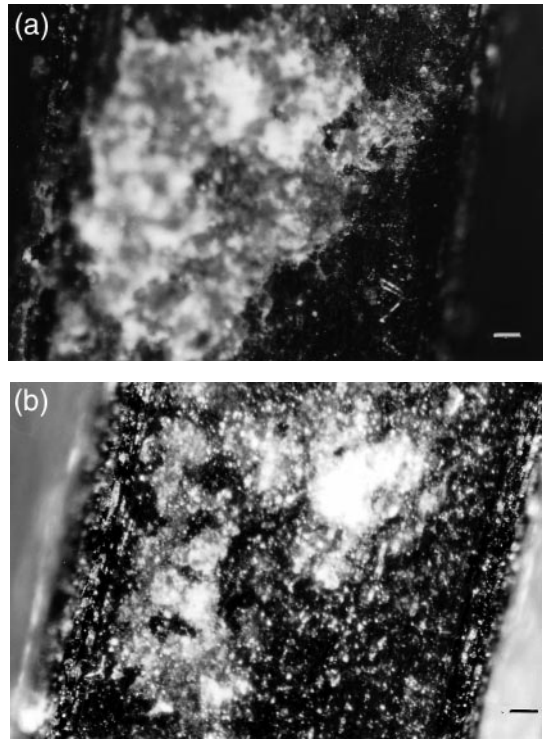

Figure 1 (a,b) Reflected light images of retrieved NiTi wires demonstrating accumulation of microcrystalline precipitates. (Brightfield, ×50 original magnification, bar = 100 μm.)

Results

Optical microscopic investigations showed that the surfaces of the retrieved specimens were partially covered by islands of an amorphous material with regional accumulation of micro-crystalline particles exhibiting a birefringence effect under polarized light (Figure 1a,b). Representative micro-MIR FTIR spectra obtained from a wire surface before and after intra-oral exposure in the same patient for 1 and 3 months are presented in Figure 2. Before intra-oral exposure (reference) no peaks were found with the exception of the CO_2 atmospheric interference at 2363 cm^{-1}. Following 1 month in the oral environment (A) the following peaks were identified: –OH (3300 and 1642 cm^{-1}), N–H (3200 cm^{-1}), $–CH_3$ (2890 cm^{-1}), $–CH_2–$(2840 and 1380 cm^{-1}), CO_2 atm (2363 cm^{-1}), –COOH (1730 and 1300 cm^{-1}), –CONH– (1635 cm^{-1} amide I, 1542 cm^{-1} amide II, 1250 cm^{-1} amide III), $–CO_3$ (1450 and 870 cm^{-1}), –CH–OH (1170 cm^{-1}), and $–CH_2OH$ (1020 cm^{-1}). After 3 months *in vivo* (B) the intensities of N-H, amide I–III, and –CH–OH groups were increased, implying higher concentration of irreversibly adsorbed proteinaceous matter on the wire surface.

Figure 3 (a–c) depicts SEI of a wire in the as-received condition and following intra-oral exposure in two patients for 2 and 6 months, respectively. The specimen retrieved after 2 months (Figure 3b) was covered by a well-organized integument leaving free only small areas of exposed wire surface. These areas demonstrated important morphological variations compared with the reference specimens (Figure 3a). The striations were smoother and increased surface porosity occurred. The 6-month

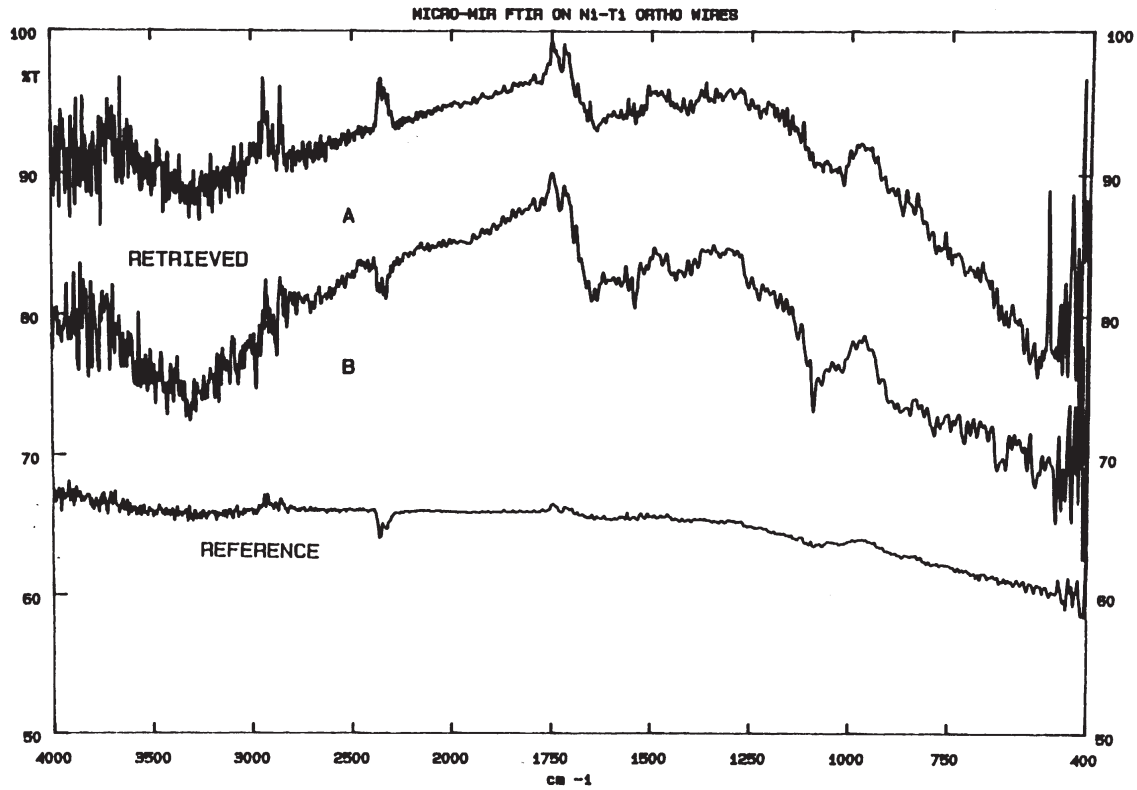

Figure 2 Micro-MIR-FTIR spectra of an as-received archwire specimen (reference) and of the same wire after intra-oral exposure to the oral cavity of the same patient for 1 (A) and 3 months (B).

retrieved specimen from another patient (Figure 3c) exhibited less surface coating from acquired biofilms, which may reflect differences in the intra-oral conditions between different individuals participating in the study. Again, wire striations were smoother and regions of plastically deformed material were identified. Porosity and pore size of the wire surface was also increased.

Figure 4 (a–d) illustrates the morphological, compositional, and elemental distribution images of a transitional zone between a free-wire surface and a surface covered by a biofilm after 2 months intra-oral placement. The SEI showed that the biofilm region was dominated by highly scattered granular particles (<10 μm in diameter) embedded in an amorphous substrate, which completely masked the topographic features of the underlying alloy surface (Figure 4a). Some large crystal aggregates were also identified. The free-wire surface demonstrated surface smoothing and increased porosity. The corresponding compositional BEI revealed a wide range of grey level at the biofilm covered region compared with the white alloy surface implying the presence of elements of lower atomic number than the wire alloy (Figure 4b). Electron probe micro-analysis demonstrated a uniform distribution of Na, K, and Cl at the low atomic number region, while the distribution of Ca and P showed well-defined high intensity islands (Figure 4c) corresponding to the high back-scattering regions of the surface covered by a biofilm. The distribution of the alloy components Ni and Ti extended beyond the alloy margins coated from the integument, indicating that the biofilm thickness was less than the mean sampling depth of the micro-analysis conditions for Ni and Ti (Figure 4d).

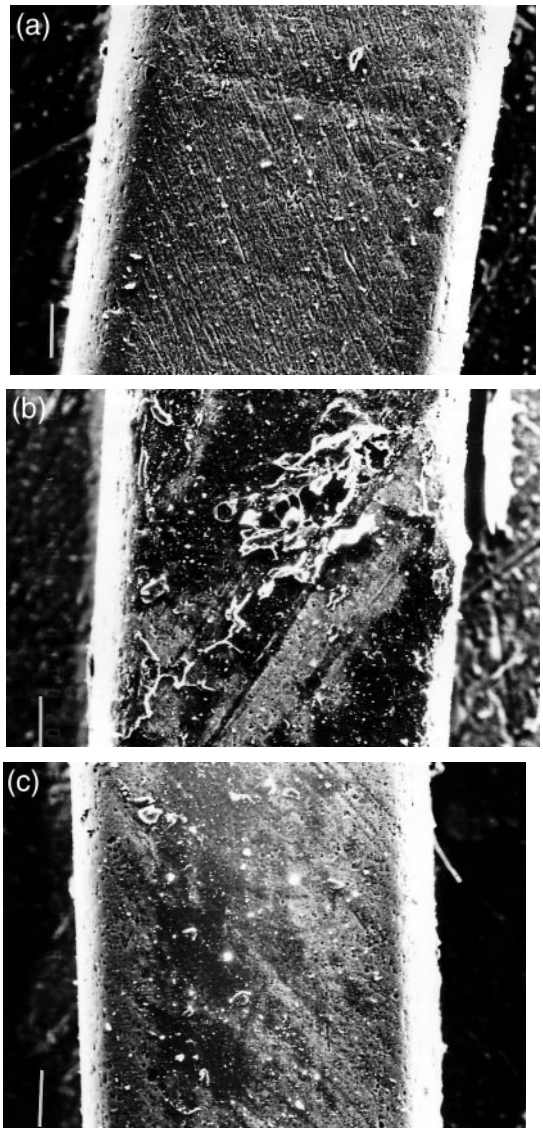

Figure 3 Secondary electron image of NiTi archwire surfaces (photographs of different samples, original magnification ×100, bar = 100 μm). (a) As-received. (b) After 2 months intra-oral exposure. (c) After 6 months intra-oral exposure.

Prolonged intra-oral exposure of the wires in the same patient resulted in the formation of well-organized and heavily calcified solid integuments relative to shorter exposure times. No difference was noted between the incisor and molar regions with respect to the extent of the changes induced; similarly, an identical ageing pattern was observed for round, square, and rectangular archwires from different manufacturers.

The microscopic investigation of the metallographically-polished specimens showed that the wire surfaces engaged in the bracket slots demonstrated material loss and various modes of corrosion such as delamination, crevices, and pitting (Figure 5a,b). The sites of the retrieved wires engaged to the brackets exhibited a much smaller grain size (Figure 6a) compared with the etched reference wires (Figure 6b).

Discussion

Retrieval analyses have recently gained special interest in dental materials due to the critical information derived from investigating the performance of the material in the environment in which it was intended to function. This method has furnished information that has not been described before, such as the attack of specific microbial species on orthodontic adhesives (Matasa, 1995). Despite its recent application in dental materials, retrieval analysis has been used for some decades in orthopaedic applications of biomaterials (Rostoker *et al.*, 1978). Recently, this approach has gained interest and a significant number of the published studies concerning orthopaedic materials deal with retrieved materials (Witkiewich *et al.*, 1996). Furthermore, the development of standards for the retrieval analysis of orthopaedic materials is strongly indicative of the significance of this method in studying the performance of materials (International Standards Organization/ Draft International Standards, 1996; American Society for the Testing of Materials, 1997). However, a disadvantage of retrieval studies pertains to the lack of a sequential description of the alterations induced and the inability to derive quantitative data.

The results of the retrieval showed that the NiTi wires were coated by intra-orally formed proteinaceous integuments that masked the alloy surface topography to an extent dependent on the individual patient's oral environmental conditions and the intra-oral exposure period. The organic constituents of the film acquired on the alloy surface were amide, alcohol, and

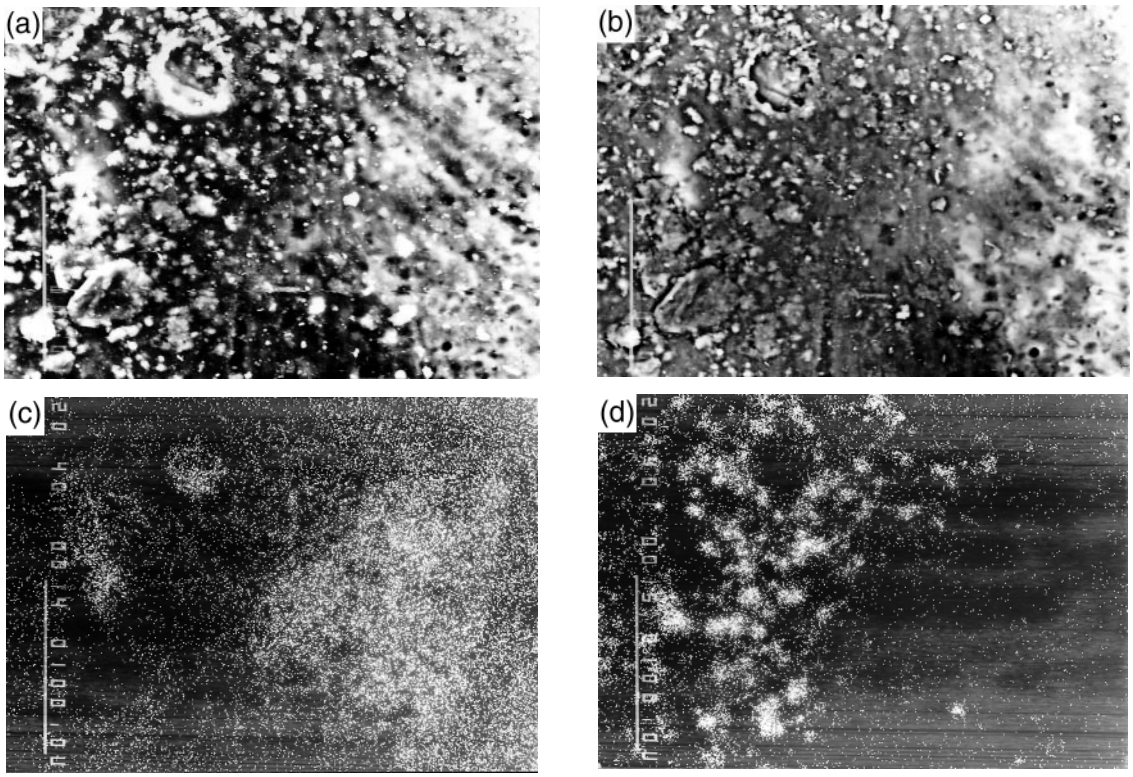

Figure 4 SEM-WDS X-ray micro-analysis of a NiTi wire specimen following 2 months of intra-oral exposure. (Original magnification ×400, bar = 100 μm.) (a) SEI. (b) BEI-COMPO. (c) X-ray image of Ni. Identical distribution was observed for Ti. (d) X-ray image of Ca. The distributions of Na, K, and P show identical patterns as Ca.

carbonate, whereas the predominating elemental species were Na, K, Cl, Ca, and P. The elemental distribution of the biofilm complies with the formation of NaCl, KCl, and Ca-P crystalline precipitates on wire surfaces.

Biofilm formation and mineralization on materials placed intra-orally are considered non-specific mechanisms. The initial steps are governed by the surface properties of the material (Baier *et al*., 1984), whereas the extent of mature plaque formation is influenced predominately by individual intra-oral conditions (Hannig, 1999). These integuments substantially modify the morphology, surface composition, and electrochemical reactivity of wires. Nevertheless, mineralized regions may provide a protective effect over the alloy substrate especially under low pH conditions where the corrosion rate of NiTi wires is increased (Oshida *et al*., 1992).

On a clinical level, the described alterations may impact on the roughness of the wire with detrimental effects on the efficacy of certain mechano-therapeutic approaches such as sliding mechanics. The implication of wire surface roughness on friction during sliding has not been unequivocally defined. The vast majority of the *in vitro* studies dealing with this issue have shown that friction increases with increased roughness of the wire and bracket surfaces (Dowing *et al*., 1994; Tselepis *et al*., 1994; Bazakidou *et al*., 1997; Ryan *et al.*, 1997). Those studies indicated that, in general, β-Ti and NiTi wires and ceramic brackets present increased friction due to their roughened surfaces during manufacture. However, it has been proposed that friction is independent of wire roughness (Kusy and Whitley, 1990). These authors measured coefficients of friction of orthodontic

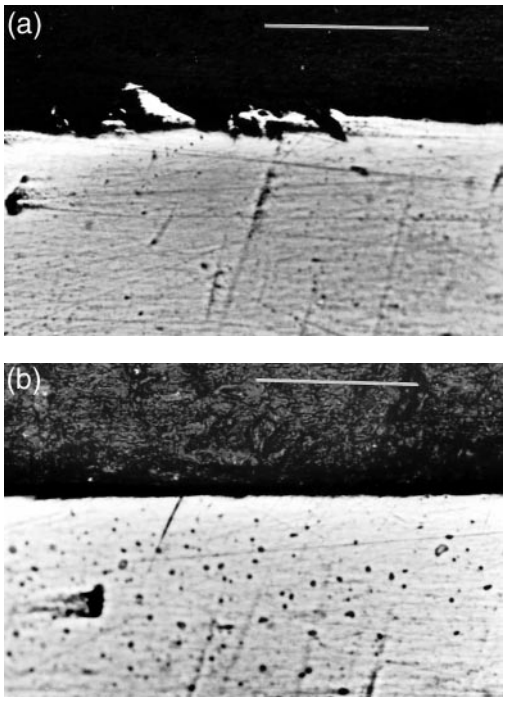

Figure 5 Reflected light image of a longitudinally sectioned and metallographically polished retrieved NiTi wire specimen after 4 months intra-oral exposure. (Original magnification ×160, bar = 100 μm.) (a) Surface demonstrating de-lamination, pitting, and crevice corrosion. This surface was engaged to the bracket slot. (b) Surface of the wire not engaged in the bracket slot. Only pitting and crevice corrosion were observed.

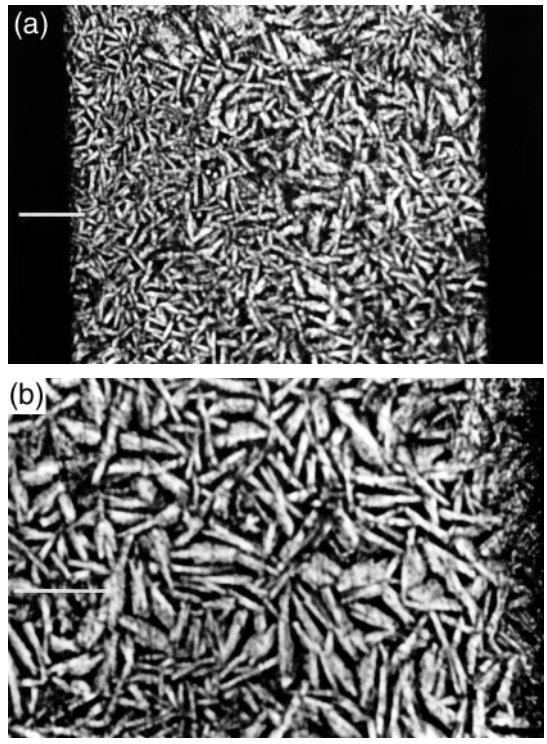

Figure 6 Reflected light image of a longitudinally sectioned, metallographically polished, and etched NiTi wire specimen. (a) Retrieved wire specimen after 5 months intra-oral exposure. Note the reduced grain size of the region engaged in the bracket (left). (Original magnification ×50, bar = 100 μm.) (b) As-received (reference) wire specimen. Grain size distribution is uniform with the exception of a small surface region demonstrating reduction in grain size possibly due to manufacturing procedures. (Original magnification ×80, bar = 100 μm.) Note: Figure 6a was taken at ×50 magnification because the region of reduced grain size extends far beyond the surface and the low magnitude allows for the differentiation of small from large grain size regions. In Figure 6b, ×80 magnification was chosen for better resolution of the thin small grain size subsurface layer produced from the manufacturing process because of the relatively small width of the region imaged. Nevertheless, in Figure 6b at ×80 magnification, the image width is approximately 85 per cent of that of Figure 6a and, therefore, this figure does not represent an isolated region.

wires with root mean square (RMS) surface roughness ranging from 0.04 (stainless steel) to 0.23 μm (NiTi) on stainless steel and polycrystalline alumina flats of 0.03 and 0.26 μm roughness, respectively. The results showed that the β-Ti wire (RMS = 0.14 μm) presented the highest friction coefficient although the NiTi was the roughest (RMS = 0.23 μm). Nevertheless, they reported mass transfer from the β-Ti to the stainless steel wire and polycrystalline alumina contact flats, a fact which implies that the lower compressive yield of β-Ti might have been implicated in the measurements. Since the friction coefficient depends on both surface roughness of involved surfaces and compressive yield strength of materials (Sears *et al*., 1982; Ashby and Jones, 1988), further clarification of the implication of roughness on friction is needed employing an experimental configuration where these two parameters will not vary simultaneously. An additional explanation is that the surface roughness differences shown may lack an effect on friction *in vivo* and greater differences may be required to produce a measurable effect on friction variants.

Moreover, the results of *in vitro* studies cannot be applied to this investigation since the frictional test by Kusy and Whitley (1990) was performed on dry and relatively clean sample surfaces, and therefore, no biofilm or calcified regions were included. The adsorption of these intra-oral integuments may strongly modify the friction coefficient by producing a boundary lubrication effect (Ashby and Jones, 1988) which may greatly reduce friction coefficient, i.e. saliva protein adsorption and plaque accumulation; alternatively calcified integuments may increase the surface roughness and resistance to shear forces.

The foregoing considerations coupled with the complexity of the oral environment may explain the results of a recently published article investigating the effect of using different wires on the rate of space closure *in vivo* (Kula *et al.*, 1998). That study showed that there was no statistical difference with respect to average space closure *in vivo* between conventional, and ion-implanted β-Ti wires, whereas the rate obtained was similar to that reported for stainless steel archwires in laboratory set ups. Thus, further *in vivo* studies are needed to resolve the effect of surface roughness on friction.

The ageing pattern described and characteristics of biofilm maturation appears dependent on exposure time, rather than archwire brand or size, since identical patterns were observed for archwires of various manufacturers and cross-sections. Similarly, there was no difference between the effects of stainless steel versus elastomeric ligatures on the wire alloy surface.

The increased extent of biofilm coating and the pronounced mineralization observed on wire surfaces placed intra-orally in the same patient for increased periods is apparently attributed to plaque maturation and mineralization. The lack of substantial differences in the extent of biofilm coating between anterior and posterior regions supports previous findings suggesting that biofilm formation occurs at similar rates at different intra-oral sites (Quirynen *et al.*, 1989).

The morphology of the free wire surfaces demonstrated distinct differences between retrieved and reference groups. Surface smoothness and increased pitting were the main findings. Although pitting corrosion occurs on retrieved NiTi wire surfaces (Sarkar and Schwaninger, 1980; Oshida *et al.*, 1992), no effects on the physical and mechanical properties of the wires have been identified (Schwaninger *et al.*, 1982). Crevice corrosion and selective nickel dissolution from the near surface region has also been documented on NiTi wires *in vitro* (Oshida *et al.*, 1992). However, such compositional changes have been reported in crevices of both as-received and used NiTi archwires (Grímsdóttir and Hensten-Pettersen, 1997) implying the possibility of manufacturing defects. The discrepancy in the results of the above studies may be attributed to the different experimental approaches followed. In the former, analysis of the near surface region of longitudinally-sectioned wire specimens was performed, whereas the latter involved spot analysis of surface located crevices that may be affected by surface topographical features.

Several important differences were noted in the surface profile morphology of the longitudinally sectioned and polished wires relative to the as-received specimens. Surface regions engaged to the bracket slot showed excessive wear, while characteristic patterns of delamination were observed. The enhanced deterioration of this specific region may be assigned to compressive forces accompanying wire activation through ligation and possible frictional damage produced inside the slot. Alternatively, this effect may arise from ploughing during sliding of NiTi alloy wire on stainless steel bracket slots. NiTi and β-Ti wire alloys are most likely to present this effect because of the presence of rough surfaces associated with the wire drawing process.

At the wire edge opposing that engaged to the bracket, increased frequency of cracks and crevices was observed, presumably due to the presence of tensile forces produced locally due to the wire engagement. These force vectors induced changes in the micro-structure of the alloy, resulting in a reduction in grain size at the compressed locations, which extended beyond the near-surface region. Changes in grain size are well documented in NiTi metallurgy as SIM (stress-induced martensite), where the martensitic transformation occurs below the transition temperature range (TTR) when external stress

is applied (Weyman, 1993). Whether this transformation reflects an effect with clinical implications possibly related to the expression of the superelastic properties of the wire is not known.

Conclusions

Retrieved NiTi orthodontic wires are characterized by the formation of a proteinaceous biofilm, the organic constituents of which are mainly alcohol, amides, and carbonate.

The irreversible formation of precipitates and the development of microcrystalline NaCl, KCl, and Ca-P deposits substantially alter the surface composition and topography of the wire alloy.

Delamination, pitting, and crevice corrosion defects, as well as reduction in the alloy grain size were identified in retrieved NiTi wires. These alterations may arise from a combination of intra-oral conditions and loading attributed to the engagement of the wire into the bracket slot.

The described alterations of retrieved wires may profoundly modify the reactivity of the wire surface with undetermined effects on the corrosion resistance, nickel dissolution, and frictional resistance of the archwires.

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