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Relationship between the metallurgical structure of experimental titanium miniscrew implants and their torsional properties

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SUMMARY The aims of this study were to investigate the torsional properties of three experimental titanium miniscrew implants for orthodontic anchorage and to determine the relationship between the torsional properties and metallurgical structures.

Experimental miniscrew implants with a diameter of 1.4 mm were fabricated from commercially pure (CP) titanium (alpha-titanium), Ti-4Al-4V (duplex alpha-beta-titanium), and Ti-33Nb-15Ta-6Zr (beta-titanium). Micro-X-ray diffraction (XRD) was performed to identify phases, and microstructures of etched cross-sections were obtained with scanning electron microscopy (SEM). Implants were loaded in torsion (n=5), and mean moments and twist angles at fracture were statistically compared using the Kruskal–Wallis and Mann–Whitney U-tests. Cyclic torsional moment for fracture of starting square wires (2×2×30 mm) was measured (n=3).

At fracture, the Ti-4Al-4V and Ti-33Nb-15Ta-6Zr implants demonstrated significantly higher mean torque than the CP titanium implant, while the Ti-33Nb-15Ta-6Zr implant had a significantly higher mean twist angle than the other two implants. The CP titanium and Ti-33Nb-15Ta-6Zr implants displayed good fatigue performance and excellent ductility. Ti-33Nb-15Ta-6Zr beta-titanium alloy is suitable for manufacturing miniscrew implants since it has excellent torsional properties.

Introduction

Anchorage control is essential for establishing ideal occlusion in orthodontic treatment. A variety of skeletal anchorage systems, using dental implants (Roberts *et al.*, 1984), palatal implants (Wehrbein *et al.*, 1999), miniplates (Umemori *et al.*, 1999), and titanium miniscrew implants (Creekmore and Eklund, 1983; Kanomi, 1997), have been introduced during the past two decades. Above all, miniscrew implants have been most popular because they are simple to place and use, cause little discomfort, and are cost-effective. However, miniscrew implant fracture is a serious problem for both orthodontists and patients because it is sometimes difficult to remove the implant fragment from inside the bone (Buchter *et al.*, 2005; Park *et al.*, 2006). Therefore, sufficient mechanical strength is needed for miniscrew implants to resist the torsional stresses developed during placement and removal.

A recent study (Iijima *et al.*, 2008) investigated the torsional properties of commercial miniscrew implants and concluded that titanium alloy miniscrew implants containing small amounts of vanadium and aluminium with a duplex microstructure (alpha-beta-titanium) had a significantly higher mean torque at failure compared with commercially pure (CP) titanium miniscrew implants with a single-phase alpha-titanium microstructure. While Ti-6Al-4V is in widespread use as a dental implant alloy because of its superior mechanical properties compared with CP titanium (Okazaki *et al.*, 1998; Koike *et al.*, 2007), possible cytotoxic and adverse tissue reactions caused by vanadium have been reported (Steinemann, 1980; Wapner, 1991; Thompson and Puleo, 1996). Consequently, development of alternative titanium alloys with improved biocompatibility should be investigated.

The purpose of this study was to investigate the effects of composition on torsional properties and microstructures of experimental titanium miniscrew implants machined from CP titanium and two titanium alloys (Ti-4Al-4V; Ti-33Nb-15Ta-6Zr). It was hypothesized that the Ti-33Nb-15Ta-6Zr implant would have comparable mechanical properties with the Ti-4Al-4V implant, which is currently the most popular miniscrew implant composition for clinical orthodontics.

Materials and methods

Materials

Experimental titanium miniscrew implants with three different compositions (CP titanium, implant A; Ti-4Al-4V, implant B; and Ti-33Nb-15Ta-6Zr, implant C) and with a diameter of 1.4 mm were manufactured (Nishimura Ltd, Sabae, Fukui, Japan) by machining the cold-worked wires, using the McLoader program. The morphological features of the experimental miniscrew implants are shown in Figure 1. Starting square wires, materials A, B, and C, for manufacturing the miniscrew implants with a cross-section dimension of $2 \times 2 \times 30$ mm were also obtained. X-ray fluorescence analyses (JSX-3200; Jeol, Tokyo, Japan) verified that the nominal compositions were (1) nearly pure titanium for implant A; (2) titanium alloy containing 4.2 per cent vanadium, 3.9 per cent aluminium, 0.2 per cent iron, and 0.03 per cent manganese for implant B; and (3) titanium alloy containing 33.2 per cent niobium, 14.7 per cent tantalum, 6.1 per cent zirconium, and 0.2 per cent iron for implant C.

Micro-X-ray diffraction

Micro-X-ray diffraction (XRD) analyses (Rint-2000; Rigaku, Tokyo, Japan) were performed at room temperature with CuKα radiation at 40 kV and a tube current of 300 mA to identify the phases in the three experimental titanium miniscrew implants. The sample stage was fixed at 25 degrees to the incidence angle of the X-ray and rotated from −120 to +120 degrees about the φ-axis to minimize the effect of preferred orientation. The locations of these axes have been reported previously (Iijima *et al.*, 2002). The micro-XRD spectra were obtained from the longitudinal surfaces near the central area of the miniscrew implants. A 50 μm diameter collimator was used to establish the dimensions of the analysis area. The diffracted X-rays were detected by a position-sensitive proportional counter. The measurement time was 10 minutes.

Scanning electron microscope observations

Each specimen was encapsulated in epoxy resin (Epofix; Struers, Copenhagen, Denmark) and then polished using a series of silicon carbide abrasive papers and a final slurry of 0.05 μm alumina particles. The polished surfaces were etched in a solution of 2.5 per cent HF, 2.5 per cent HNO_3, and 95 per cent H_2O (Kroll's reagent) for 10–20 seconds. The specimens were sputter coated with gold and then observed with an accelerating voltage of 15 kV in a vacuum using a scanning electron microscope (SEM) (X-650; Shimadzu, Kyoto, Japan).

Torsional test procedures

A custom-fabricated device for torsional loading of the miniscrew implants is shown schematically in Figure 2. The head portion of each miniscrew implant was removed using a water-cooled diamond saw (Isomet; Buehler, Lake Bluff, Illinois, USA) before insertion of the specimen into the grips. The torsional moment, obtained as the angle of rotation (twist angle), was increased at 90 degrees/minute until fracture of the miniscrew occurred ($n=5$). The cyclic torsional moment for a range of rotation angles from −45 to +45 degrees was also measured until fracture occurred ($n=3$) for the starting square wires.

Statistical analysis

Statistical analysis was performed with Statistical Package for Social Science (version 14.0J for Windows; SPSS Inc., Chicago, Illinois, USA). Descriptive statistics, including the mean, standard deviation, median, minimum and maximum values of the torque, and twist angle at fracture, were calculated for the three groups. The Kolmogorov–Smirnov normality test and the Levene variance homogeneity test were first applied to the data. Because the data were not normally distributed, a Kruskal–Wallis test was used to determine whether significant differences existed among the groups. The Mann–Whitney *U*-test was then used for two independent groups, and the Bonferroni correction was applied.

Results

Representative micro-XRD patterns from the manufactured experimental miniscrews are shown in Figure 3. The pattern

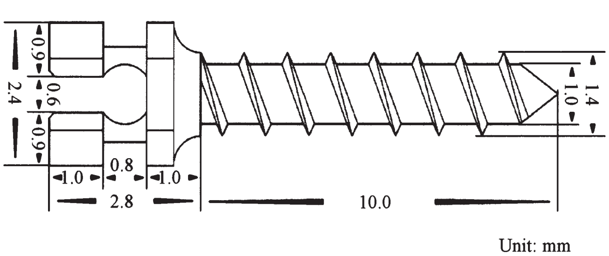

Figure 1 Morphological features of the experimental titanium miniscrew implants.

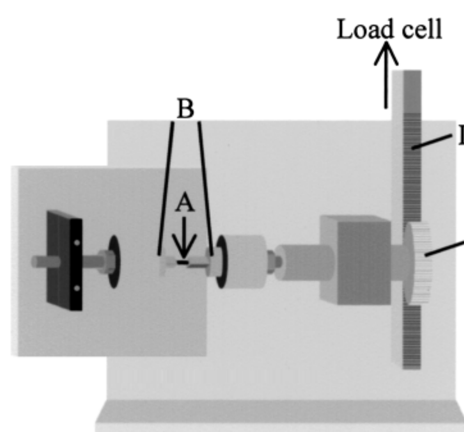

Figure 2 Custom-fabricated device for torsional testing. A, miniscrew implant specimen; B, jaws that hold implant specimen; C, pinion that engages the rack; D, rack attached to the moving crosshead of the mechanical testing machine.

for implant A contained three peaks, which could be indexed to the 100, 101, and 110 crystal planes of alpha-titanium. The pattern for implant B contained eight major peaks (100, 002, 101, 102, 110, 103, 112, and 201) for alpha-titanium. In contrast, the pattern for implant C contained five peaks, which could be indexed to the 110, 200, 211, 220, and 310 crystal planes of beta-titanium. The matches of the interplanar spacings (d-spacings) obtained from these peaks were in excellent agreement with the data of the International Center for Diffraction, Swarthmore, Pennsylvania, USA powder standards for alpha-titanium (No. 44-1294) and beta-titanium (No. 44-1288).

Representative photomicrographs of the cross-sectioned miniscrew implants are shown in Figure 4. The microstructure of implant A was single phase with equiaxed grains of alpha-titanium. Implant B had a two-phase microstructure, containing small globular particles of beta-titanium in a matrix of alpha-titanium. The fine-scale alpha-titanium grains were difficult to resolve, and the volume percentage of beta-titanium was insufficient for detection by micro-XRD. The microstructure of implant C was single phase, with equiaxed grains of beta-titanium. The beta-titanium grain size for implant C was smaller than the alpha-titanium grain size for implant A.

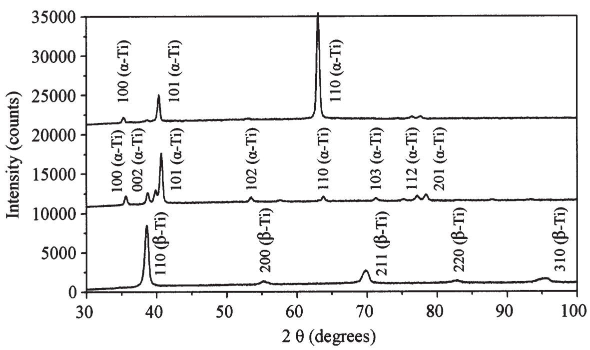

Figure 3 Micro-X-ray diffraction patterns from three different experimental miniscrew implants. α-Ti, alpha-titanium; β-Ti, beta-titanium. Implant A, upper; implant B, middle; implant C, lower.

Mean torsional moment (kgf•cm) and twist angle (degrees) at fracture are summarized for the experimental miniscrew implants in Table 1. From the Kruskal–Wallis and Mann–Whitney *U*-tests, implants B (1.94 ± 0.04 kgf•cm) and C (1.80 ± 0.11 kgf•cm) showed significantly higher mean torque at fracture than implant A (1.23 ± 0.07 kgf•cm). For mean twist angle at fracture, implant C had a significantly higher value (608.7 ± 114.5 degrees) than the other implants (267.4 ± 126.9 degrees for implant A and 76.6 ± 22.6 degrees for implant B).

The results of the cyclic torsional test for the starting square (2 × 2 × 30 mm) wires are shown in Figure 5. Material B used for fabrication of implant B (Ti-4Al-4V) fractured after only two to seven torsional cycles, although it showed higher mean torque at failure (9.21 kgf•cm). Although materials A and C, used for fabrication of implants A and C, showed a lower mean torque at fracture (4.12 and 7.04 kgf•cm, respectively), an increased number of (approximately 100) cycles were required for failure.

Discussion

It is common to separate titanium and its alloys into four categories: (1) unalloyed, (2) alpha and near-alpha, (3) alpha-beta, and (4) metastable beta, referring to the phases

Table 1 Mean and standard deviation for torque values and twist angle at fracture of the groups of three different experimental miniscrew implants.

Experimental implant group	Torque value (kgf•cm)	Twist angle (°)
Implant A	1.23 ± 0.07[a]	267.4 ± 126.9[c]
Implant B	1.94 ± 0.04[b]	76.6 ± 22.6[d]
Implant C	1.80 ± 0.11[b]	608.7 ± 114.5[e]

Identical letters indicate that mean values are not significantly different ($P > 0.05$, Bonferroni correction for multiple comparison). Implants B and C showed a significantly higher mean torque values at fracture than Implant A. Implant C showed a significantly higher mean twist angle at fracture than the other implants.

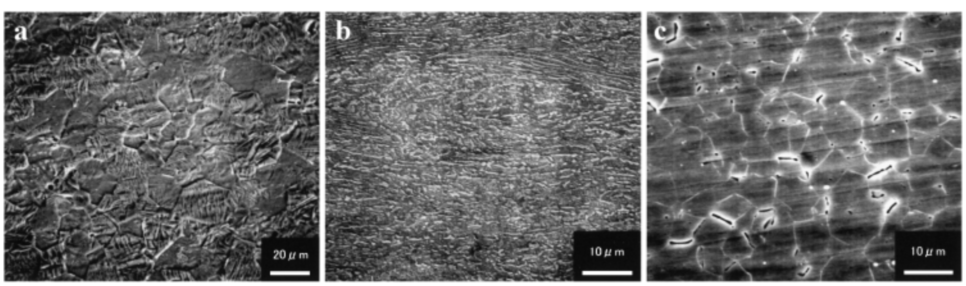

Figure 4 Scanning electron photomicrographs of three different experimental miniscrew implants. (a) Implant A (original magnification, ×600), (b) implant B (original magnification, ×1500), and (c) implant C (original magnification, ×1500). Equiaxed alpha-titanium was found in implant A. The microstructure of implant B contained small globular particles of beta-titanium in a matrix of alpha-titanium. Equiaxed beta-titanium was found in implant C.

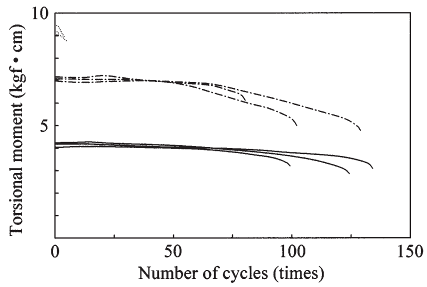

Figure 5 Cyclic torsional test (from −45 to +45°) of the square wire with 2 × 2 × 30 mm dimensions until fracture. Material A, continuous line (bottom plots); material B, dotted line (top plots); material C, dotted and dashed line (middle plots). Material B (Ti-4Al-4V) fractured after a very small number of torsion cycles although it showed higher torsional moment values. Although materials A and C showed lower torque values at fracture, they resisted cyclic torsion until approximately 100 cycles.

normally present (Donachie, 2000). Unalloyed titanium, generally known as CP titanium, has a lower strength than titanium alloys, and the primary difference between the different grades of CP titanium is oxygen and iron content. Alpha alloys contain relatively high concentrations of alpha stabilizers (such as aluminium, tin, and zirconium) and low concentrations of beta stabilizers (such as molybdenum, vanadium, tantalum, niobium, manganese, iron, chromium, cobalt, nickel, copper, and silicon). Alpha-beta alloys contain one or more alpha stabilizers or alpha-soluble elements plus one or more beta stabilizers in larger concentrations than in the near-alpha alloys. The inclusion of a small amount of beta-titanium, or an entirely beta structure, permits alpha-beta alloys to be strengthened by solution heat treatment and ageing. The metastable beta alloys are richer in beta stabilizers and lower in alpha stabilizers than alpha-beta alloys. The principal advantages of metastable beta alloys are that they have high hardness, excellent forgeability, and good cold formability (Donachie, 2000). Beta-titanium alloys in the heat-treated condition (single phase) have good ductility and toughness but relatively low strength.

In the present study, the microstructural results from micro-XRD analyses and SEM observations, along with the compositions confirmed by X-ray fluorescence analysis, showed that implant B (Ti-4.2V-3.9Al-0.2Fe-0.03Mn) had a duplex alpha-beta structure and that implant C (Ti-33.2Nb-14.7Ta-6.1Zr-0.2Fe) had a single-phase beta-titanium structure. These results were consistent with known composition–microstructure relationships for titanium alloys (Donachie, 2000).

Sufficient mechanical strength is required for miniscrew implants to resist torsional stresses that are developed at the screw threads during clinical placement or removal. The strength of CP titanium or a titanium alloy miniscrew implant depends on the microstructure, which is influenced by composition, heat treatment, and the machining process (Donachie, 2000; Niinomi, 2003; Banerjee *et al.*, 2004).

Although fracture at the neck of the orthodontic miniscrew implant is often experienced under clinical conditions because of the effect of stress concentration, it is largely influenced by the design of the implant. Because this study focused on the relationships between microstructures and torsional performance rather than the design (morphological) factor, the three groups of experimental implants had the same design with a 1.4 mm diameter. A recent study (Iijima *et al.*, 2008) concluded that commercial miniscrew implants made of alpha-beta titanium alloys had superior torsional properties compared with CP titanium miniscrew implants. However, the commercial titanium alloy miniscrew implants contained small amount of vanadium, which may cause cytotoxic and adverse tissue reactions (Kravitz and Kusnoto, 2007).

The present study investigated the torsional properties of an experimental Ti-33Nb-15Ta-6Zr implant as an alternative titanium miniscrew alloy because niobium, tantalum, and zirconium show excellent biocompatibility (Okazaki and Gotoh, 2005). In addition, Rose *et al.* (1998) reported that beta-titanium alloy (Ti-11Zr-6.5Zn-4.5Sn) had no effect on the rate of cell proliferation, suggesting that beta-titanium alloy might have low toxicity. The mean torque values at fracture for implants B and C were significantly higher than for the experimental pure titanium miniscrew implant (implant A). Moreover, the mean twist angle at fracture of implant C was significantly higher than for the other two experimental implants. In addition, cyclic torsion testing of the starting square wire specimens (2 × 2 × 30 mm dimensions), used for machining the experimental implants, showed that the alpha-beta alloy (material B) fractured after only a very small number of torsion cycles, whereas the pure titanium specimen (material A) and beta-titanium specimen (material C) resisted approximately 100 torsional cycles, suggesting that both implants A and C have good fatigue performance along with excellent torsional ductility. The ability to resist high torque moment is essential for a miniscrew implant during placement or removal, and material B (9.21 kgf•cm) showed higher mean torque at fracture than materials A and B (4.12 and 7.04 kgf•cm, respectively). In addition, high ductility is an important feature because an excessive lateral force sometimes occurs at a miniscrew implant when brushing teeth and tying ligature wires. If such excessive force occurs, miniscrew implants with high ductility may be deformed permanently without fracture, and this should be a clinical advantage provided that the amount of permanent deformation is very small.

Conclusion

Ti-33Nb-15Ta-6Zr beta-titanium alloy is suitable for manufacturing commercial miniscrew implants since it has excellent torsional mechanical properties.

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