# Rate of Pull-out Strength Gain of Dual-Etched Titanium Implants: A Comparative Study in Rabbits

David Baker, DDS, MSD\*/Robert M. London, DDS\*\*/Robert O'Neal, DDS\*\*\*

The purpose of this study was to investigate the rate of pull-out strength gain of an etched titanium implant surface. Rabbit tibiae were used to compare machined titanium and proprietary dual-etched titanium implants. Two custom cylindric implants (3 mm in diameter and 4 mm in length) were placed in each right anteromedial tibia in 31 rabbits. At weeks 1, 2, 3, 4, 5, and 8, the implants in 5 rabbits were subjected to failure shear loading in a pull-out test. For shear failure testing, each tibial segment was mounted in a precision alignment jig, and an Instron pull-out test was performed on each implant. Beginning at week 3, there was a statistically significant difference (P < .01) between the dual-etched and the machined implants. There was a significant increase in strength for dual-etched implants between week 5 and week 8, while the machined implants did not show an increase during this time interval. The etched implants maintained a significantly greater pull-out strength for the remainder of the study, with a 3.2-fold greater mean strength at 8 weeks, equivalent to 6 months in humans. At 3 weeks, the etched implant's strength exceeded the strength that the machined implant had achieved at 8 weeks. In short-term healing in the rabbit tibia, the dual-etched surface demonstrated a more rapid rate of pull-out strength gain than the machined surface and remained significantly stronger throughout the 8 weeks of the study.

(INT J ORAL MAXILLOFAC IMPLANTS 1999;14:722-728)

Key words: dual-etched titanium implants, healing speed, machined titanium implants, pull-out strength

There is clinical interest in dental implants that integrate faster. The term *integration strength* refers to the force required to break the bond between the implant and the bone. Integration strength may be of particular value in areas in which there is an increased risk of failure because of poor bone quality. In these areas, the increased integration strength may add to clinical predictability by counteracting the bone strength deficit. The rate of integration strength gain is also of interest. Clinicians use arbitrary healing time intervals, uncertain of what minimum time period is needed for integration. Earlier strength may reduce integration intervals, speeding patient care. Early stability and strength may prevent implant loss resulting from overload. With increasing use of single-stage implant surgery, greater early strength may also reduce concerns regarding incidental loading of healing abutments. Additionally, this may improve the survival of implants that are subjected to heavy transmucosal loading during healing.

The 1996 World Workshop in Periodontics<sup>1</sup> concluded that the surface characteristics of an implant, particularly roughness, may direct tissue healing. However, these macro-rough surfaces could potentially cause failure of the implant because of increased bacterial aggregation on the rough surface,<sup>2</sup> or breakdown of the hydroxyapatite.<sup>3,4</sup>

Several investigators have grit blasted implant surfaces to achieve a roughened surface. A dog study has shown that titanium oxide grit blasting leads to a higher percentage of bone contact when

<sup>\*</sup>Private Practice Limited to Periodontics, Bellingham, Washington.

<sup>\*\*</sup>Associate Clinical Professor of Periodontics, University of Washington School of Dentistry, Seattle, Washington.

<sup>\*\*\*</sup>Associate Professor and Director of Advanced Periodontics, University of Washington School of Dentistry, Seattle, Washington.

Reprint requests: Dr Robert M. London, 7331 W. Mercer Way, Mercer Island, WA 98040. Fax: (206) 230-8157. E-mail: rlondon@u.washington.edu

COPYRIGHT © 2000 BY QUINTESSENCE PUBLISHING CO, INC. PRINTING OF THIS DOCUMENT IS RESTRICTED TO PERSONAL USE ONLY. NO PART OF THIS ARTICLE MAY BE REPRODUCED OR TRANSMITTED IN ANY FORM WITH-OUT WRITTEN PERMISSION FROM THE PUBLISHER.

compared to a machined surface.<sup>5</sup> This grit blasting can lead to a decrease in the fatigue strength of the implant.<sup>6</sup> The process of grit blasting can leave metallurgical point defects in an otherwise homogeneous matrix.

Grit blasting followed by acid etching has produced surface characteristics resulting in the greatest bone contact percentage of the metal surfaces tested. This appears superior to grit blasting alone and to acid-pickled and titanium plasma-sprayed surfaces.<sup>7</sup> Grit particles can remain impregnated in the implant material, and are potentially a causative agent in observed tissue breakdown.<sup>8</sup> A new surface that produces a micro-roughness similar to the blasted/etched surface but uses only special high-temperature dual acid etching without grit blasting has been developed. The purpose of this dual etching is to produce a micro-rough surface that provides rapid osseointegration, while maintaining the long-term success associated with a machined implant surface.

The hard tissue-implant interface depends greatly on the quality and quantity of the oral bone. These characteristics vary in different locations within the oral cavity. The use of extraoral bone provides a consistent experimental source to compare the short-term healing of implant surfaces. Wennerberg et al showed that after 12 weeks of healing in the rabbit tibia, the rougher grit-blasted surface required higher removal torques than did a turned, machined surface.<sup>9</sup> A follow-up study compared a turned, machined surface with a 25-µm or a 75-µm aluminum oxide particle-blasted surface. That study found greater bone-to-implant contact in the 75-µm blasted surface.<sup>10</sup> In a 1-year followup, removal torques remained higher for the rougher surfaces.<sup>11</sup> Very rough surfaces were also explored, and blasting with either 25-µm or 250µm particles showed that the highly increased surface roughness was a disadvantage for bone-toimplant contact.<sup>12</sup> A comparison of niobium (micro-rough) and titanium (smooth) in rabbit bone found that niobium had higher removal torques, but otherwise there was no difference in the histomorphometric analysis in the percentage of bone contact.<sup>13</sup> Surface characteristics of electropolished implants with different oxide thicknesses and morphology were studied in rabbit cortical bone. That investigation showed the polished implants to be surrounded by less bone than rougher, machined implants, indicating that surface roughness may influence the rate of bone formation in rabbit cortical bone.<sup>14</sup> An experiment conducted in miniature pigs demonstrated that peak spacing and surface roughness were important for implant

fixation.<sup>15</sup> In that study of 3 implant surfaces, a high correlation was found between implant surface roughness and implant push-out failure load.

The rabbit is commonly used as a model to evaluate the healing of endosseous implants.<sup>16-18</sup> Functional simulation using dental implants takes place to a greater degree in large animal models<sup>5,19–21</sup>; however, initial healing of endosseous implants can be adequately evaluated in the rabbit model. Remodeling dynamics around implants were studied in 4 species, including rabbits, and it was determined that rigid osseous integration depends on a sustained elevation of remodeling activity adjacent to the bone-implant surface.<sup>19</sup> The study showed remarkably similar patterns in all the investigated species, indicating that rabbits are an appropriate model to investigate healing of endosseous implants. Ericsson et al studied titanium oxide-blasted implants in the dog maxilla<sup>5</sup> and noted similar bone contact at 2 months compared to machined surfaces. Improved bone contact percentage at 4 months was seen only on the roughened surface.

The present study examines early healing patterns in a rabbit cortical bone model. Most prior studies have used an arbitrary time interval at which to terminate the study. There are no previous data to show what time periods may be appropriate. It is necessary to determine the minimum amount of time needed to achieve an adequate amount of osseointegration strength prior to loading of the implant. While the strength necessary to resist movement is only one factor in determining integration success, achieving strength early could reduce integration intervals or improve success rates when force levels early on might break the bond on implants slower to gain strength. This study seeks to determine the time variations in developing pull-out strength in implants of machined and dual-etched surfaces.

### Materials and Methods

Implant Design and Placement. The model utilized rabbit tibiae to compare machined and dual-etched titanium surfaces. Custom cylindric implants 3.3 mm in diameter and 4 mm long were utilized (machined and Osseotite surface, Implant Innovations, Palm Beach Gardens, FL) (Fig 1). External dimensions were identical and the walls were parallel. Slightly oversized (3.4 mm) cover screws were placed, and the implants were seated with the cover screw undersurfaces flush to the bone, thus assuring consistent depth of placement. Two implants were surgically placed in the right proximal tibia in a sterile surgical setting (Figs 2 and 3).

COPYRIGHT © 2000 BY QUINTESSENCE PUBLISHING CO, INC. PRINTING OF THIS DOCUMENT IS RESTRICTED TO PERSONAL USE ONLY. NO PART OF THIS ARTICLE MAY BE REPRODUCED OR TRANSMITTED IN ANY FORM WITH-OUT WRITTEN PERMISSION FROM THE PUBLISHER.



Figs 1a and 1b Scanning electron micrographs of the machined (*left*) and dual-etched (*right*) surfaces.



Fig 2a Surgical placement of experimental implant.



Fig 2b Implants are in place with mounts still attached.



Fig 3 The 3.3-mm implants are placed with oversize (3.4-mm) cover screws.

A standard drilling sequence of round bur, 2-mm twist drill, and 3-mm twist drill was used at 1500 rpm. A 3.3-mm tri-flute final bur was used for placement of the 3.3-mm-diameter implants. The implants extended through the cortical plate but did not engage the opposite cortical plate. Each implant type was alternately placed in either the proximal or more distal osteotomy. All machined and dual-etched implants were a tight press-fit initially and were seated by firm digital pressure or a light tapping force. At weeks 1, 2, 3, 4, 5, and 8 the implants in 5 rabbits were subjected to failure shear loading in the pull-out test. Eight weeks was considered adequate in this model to achieve mature integration, and closer intervals were selected to differentiate early variations.

The surgical procedure was performed on 31 New Zealand white rabbits weighing between 5

COPYRIGHT © 2000 BY QUINTESSENCE PUBLISHING CO, INC. PRINTING OF THIS DOCUMENT IS RESTRICTED TO PERSONAL USE ONLY. NO PART OF THIS ARTICLE MAY BE REPRODUCED OR TRANSMITTED IN ANY FORM WITH-OUT WRITTEN PERMISSION FROM THE PUBLISHER.



**Fig 4a** A precision alignment jig is used to ensure perpendicular implant positioning.



Fig 4b Tibia prepared for pull-out testing on the Instron machine.

and 8 pounds, sequentially treated in Central Animal Surgery at the University of Washington. The number of animals was determined by the statistical model used. A minimum number of animals per time period was selected while assuring statistical significance could reasonably be demonstrated. The protocol was approved by the university committee for animal experimentation prior to commencement of the study.

Each rabbit had anesthesia induced with intramuscular injection of xylazine (Rompin) 3 mg/kg and ketamine 35 mg/kg. Each animal was then intubated, and anesthesia was maintained with isofluorane (2% to 4%). Following surgery, each animal was given a subcutaneous analgesic of buprenorphine (Buprenex), 0.05 mg/kg. This was repeated as necessary. After the appropriate healing, the animals were sacrificed using a lethal dose of pentobarbital.

Immediately after sacrifice, each specimen was subjected to a pull-out test. The tests were performed on fresh, moist, room temperature specimens. Each test was performed within minutes of the paired implant in the same specimen. The same side was used first in each specimen. This alternated the sequence of surface types as each implant type was alternated distal or proximal with each rabbit surgery. For shear failure testing, the fresh tibia segment was mounted in a precision alignment apparatus to apply tensile forces along the long axis of the implant (Fig 4a). The segments were stabilized with a small amount of quick-setting plaster, and the pull-out test was performed to determine shear failure load of each implant. The pull-out test was performed on a standard mechanical Instron machine (Instron, Canton, MA, Model #TTMBL) (Fig 4b). After calibrating

the load cell with a precision 10 load, the machine was set to pull on a special abutment cylinder at a slow, constant rate (0.05 mm/min). The pull-out force was measured electronically by the calibrated load cell, which recorded the peak force before failure. Forces were recorded to the nearest 0.1 N. Accuracy was deemed  $\pm$  0.5 N for the load cell and electronic multiplier, which yielded a range of 0 to 500 N.

Statistics. A paired Student's t test was used to evaluate the differences in pull-out failure loads between each implant surface at each time period. Each comparison was done in the same animal with the same healing time. Each test compared the machined implant to the dual-etched implant in the same bone segment.

# Results

The mean values obtained for the maximum pullout forces for each implant surface are recorded in Table 1 and Fig 5. One machined implant failed to integrate at week 4, resulting in a reduced number of compared points (n = 4) for that time interval.

One animal was euthanized immediately after surgery because of anesthetic-related hyperactivity that resulted in a leg fracture. This animal was replaced in the study. One other rabbit developed a subcutaneous infection, but this did not affect the healing of the underlying implants.

At week 1, the bone surrounding the implants (dual-etched and machined) had just started to remodel, so the initial force was higher in this dense cortical bone. The tight initial fit appeared to persist through this interval. The dual-etched implants did require a higher average force (57.6

COPYRIGHT © 2000 BY QUINTESSENCE PUBLISHING CO, INC. PRINTING OF THIS DOCUMENT IS RESTRICTED TO PERSONAL USE ONLY. NO PART OF THIS ARTICLE MAY BE REPRODUCED OR TRANSMITTED IN ANY FORM WITH-OUT WRITTEN PERMISSION FROM THE PUBLISHER.

N), compared to the machined surface (46.7 N), but this was not significantly different.

At week 2, the dual-etched and the machined implants maintained forces similar to week 1 (58.8 N and 39.7 N, respectively).

At week 3, there was a statistically significant difference (P < .01) between the dual-etched and the machined implant. The mean force required for the machined implants had dropped to 33.3 N, while the average for the dual-etched implants was 56.5 N. This significant difference was continued at weeks 4, 5, and 8.

The dual-etched implant had a gradual increase in shear force beyond week 3. There was a significant increase in pull-out force for dual-etched implants between week 5 and week 8, while the machined implant did not show any significant increase during these later weeks. The only implant that failed to integrate was a machined implant (pull-out force = 7.6 N, which was considered to be minimal frictional loading only). That data pair was excluded, resulting in 4 data points at week 4. With 4 points, the data were still significant.

Table 1 Mean Pull-out Values in Newtons		
Integration Time	Dual-etched	Machined
Week 1	57.6 ± 24.4	$46.7 \pm 4.4$
Week 2	58.8 ± 19.6	39.7 ± 15.0
Week 3	56.5 ± 12.1	33.3 ± 3.2
Week 4	65.9 ± 16.0	$24.7 \pm 11.5$
Week 5	$71.8 \pm 16.6$	$29.2 \pm 9.3$
Week 8	114.6 ± 19.1	35.7 ± 6.6

# Discussion

The micro-rough dual-etched surface tested had greater pull-out strength than the machined implant, which is statistically significant at week 3 and beyond. It is interesting to examine the decrease in pull-out strength of the machined implant during the first 4 weeks. This is likely the result of the initial stages of bone remodeling occurring at the implant surface. The initial osteoclastic activity removes the bone that was against the press-fit implant. The bone subsequently begins to remodel. During the same time period, the dualetched surfaced implant is able to maintain the pullout force. The micro-roughness of the dual-etched surface provides an environment that gains in the pull-out force required, and the rate of strength gain can compensate for the strength loss during remodeling. Once the remodeling is completed, there is a significant increase in pull-out force resistance. The machined surfaces demonstrated a mean decrease in strength during remodeling, and at the end of 8 weeks they did not regain the initial wedged press-fit strength found at week 1.

The ideal implant surface will integrate with enough strength and rapidity to maximize integration potential without sacrificing long-term success. The surface should have a microscopic architecture conducive to cellular adhesion and translocation, but if exposed can be easily maintained. Exposure of the coronal portion of the implant is more likely because of the increasingly frequent placement of implants into anatomically compromised sites. The smoother machined surfaces may be easier to maintain in the case of exposure. Excessively rough or convoluted surfaces may enhance bacterial adhesion and are more difficult to detoxify.



Fig 5 Pull-out strength of dual-etched versus machined implants, by week.

COPYRIGHT © 2000 BY QUINTESSENCE PUBLISHING CO, INC. PRINTING OF THIS DOCUMENT IS RESTRICTED TO PERSONAL USE ONLY. NO PART OF THIS ARTICLE MAY BE REPRODUCED OR TRANSMITTED IN ANY FORM WITH-OUT WRITTEN PERMISSION FROM THE PUBLISHER. In this model, the machined surface took 4 weeks to begin showing signs that remodeling resulting in strength gain was occurring. During the same time period, the dual-etched surface was able to retain strength during the first 3 weeks and began showing a significant increase in strength at week 4.

Various techniques have been used to examine the strength of integration of a given implant surface. Commonly used are removal torque, pushout, or pull-out tests. Because of the delicate nature of the implant interface, extreme care must be taken to avoid affecting the surface interface prior to the actual test. Torque tests are commonly made using a hand-held device that can transmit variable forces during the removal. The large device can cause tipping forces that are not isolated along the long axis of the implant. While a torque test can measure the strength of screw-shaped implant interfaces, the implant thread design or apical selftapping features can influence the results. A cylindric model can be used to eliminate the variability associated with threads and remove their influence on the initial mechanical stability. Push-out studies work well on cylindric implants, provided that the material passes entirely through the bone; otherwise, apical bone must be removed, potentially disturbing the implant. Pull-out studies require careful orientation of the implant to the direction of pull to avoid inappropriate application of force. Alignment is critical, but once achieved it allows a test to evaluate only the differences in implant surfaces. This would appear to be the purest test of the surface as the only variable.

This paper attempts to identify an implant surface-bone interface that gains bond strength more rapidly than that achieved with a machined surface but that does not have a macro roughness that may contribute to difficult maintenance. Studies are needed to evaluate whether this dual-etched micro-rough surface has an advantage compared to the macro-rough titanium plasma-sprayed and hydroxyapatite surfaces in terms of bacterial response and maintainability.

#### Conclusion

The etched surface under investigation displayed pull-out strength at an early time point and achieved a high level of such strength. The dualetched surface treatment may enhance the interface strength achieved with machined-surface implants without the use of grit blasting or plasma spraying. This may preserve the purity of the surface by avoiding contaminants and may maintain the metal's inherent strength by avoiding microfractures from blasting. This method, which involves high-temperature dual acid etching, may provide a micro-rough surface with both the benefits of rapid, strong integration as reported with plasmasprayed titanium and hydroxyapatite, and the maintainability of a smooth machined surface.

## Acknowledgment

Special thanks to Dr Jack Nichols for help with the design and calibration of the Instron apparatus and to Randy Goodman for precision machining services. The staff of the University of Washington Veterinary Services were invaluable in helping with the surgical procedures. Materials were provided by 3i/Implant Innovations, West Palm Beach, Florida. Dr London provides consulting services for 3i.

#### References

- 1. Consensus report. Implant therapy I. Ann Periodontol 1996;1(1):792–795.
- Quirynen M, Bollen CM. The influence of surface roughness and surface-free energy on supra- and subgingival plaque formation in man. A review of the literature. J Clin Periodontol 1995;22(1):1–14.
- Weyant RJ, Burt BA. An assessment of survival rates and within-patient clustering of failures for endosseous oral implants. J Dent Res 1993;72(1):2–8.
- Wheeler SL. Eight-year clinical retrospective study of titanium plasma-sprayed and hydroxyapatite-coated cylinder implants. Int J Oral Maxillofac Implants 1996;11(3): 340–350.
- Ericsson I, Johansson CB, Bystedt H, Norton MR. A histomorphometric evaluation of bone-to-implant contact on machine-prepared and roughened titanium dental implants. A pilot study in the dog. Clin Oral Implants Res 1994;5(4):202–206.
- Eberhardt AW, Kim BS, Rigney ED, Kutner GL, Harte CR. Effects of precoating surface treatments on fatigue of Ti-6Al-4V. J Appl Biomater 1995;6(3):171–174.
- Buser D, Schenk RK, Steinemann S, Fiorellini JP, Fox CH, Stich H. Influence of surface characteristics on bone integration of titanium implants. A histomorphometric study in miniature pigs. J Biomed Mater Res 1991;25(7):889–902.
- Darvell BW, Samman N, Luk WK, Clark RK, Tideman H. Contamination of titanium castings by aluminum oxide blasting. J Dent 1995;23(5):319–322.
- Wennerberg A, Albrektsson T, Andersson B, Krol J. A histomorphometric and removal torque study of screw shaped titanium implants with three different surface topographies. Clin Oral Implants Res 1995;6:24–30.
- 10. Wennerberg A, Albrektsson T, Lausmaa J. Torque and histomorphometric evaluation of c.p. titanium screws blasted with 25- and 75-microns-sized particles of  $Al_2O_3$ . J Biomed Mater Res 1996;30(2):251–260.
- Wennerberg A, Ektessabi A, Albrektsson T, Johansson C, Andersson B. A 1-year follow-up of implants of differing surface roughness placed in rabbit bone. Int J Oral Maxillofac Implants 1997;12(4):486–494.

COPYRIGHT © 2000 BY QUINTESSENCE PUBLISHING CO, INC. PRINTING OF THIS DOCUMENT IS RESTRICTED TO PERSONAL USE ONLY. NO PART OF THIS ARTICLE MAY BE REPRODUCED OR TRANSMITTED IN ANY FORM WITH-OUT WRITTEN PERMISSION FROM THE PUBLISHER.

- 12. Wennerberg A, Albrektsson T, Andersson B. Bone tissue response to commercial pure titanium implants blasted with fine and coarse particles of aluminum oxide. Int J Oral Maxillofac Implants 1996;11(1):38–45.
- Johansson CB, Albrektsson T. A removal torque and histomorphometric study of commercially pure niobium and titanium implants in rabbit bone. Clin Oral Implants Res 1991;2(1):24–29.
- 14. Larsson C, Thomsen P, Lausmaa J, Rodahl M, Kasemo B, Ericson LE. Bone response to surface modified titanium implants: Studies on electropolished implants with different oxide thicknesses and morphology [published erratum appears in Biomaterials 1995 Mar;16(5):423]. Biomaterials 1994;15(13):1062–1074.
- Wong M, Eulenberger J, Schenk R, Hunziker E. Effect of surface topology on the osseointegration of implant materials in trabecular bone. J Biomed Mater Res 1995:29(12):1567–1575.
- Piattelli A, Piattelli M, Romasco N, Trisi P. Histochemical and laser scanning microscopy characterization of the hydroxyapatite-bone interface: An experimental study in rabbits. Int J Oral Maxillofac Implants 1994;9(2):163–168.

- 17. Piattelli A, Trisi P, Passi P, Piattelli M, Cordioli GP. Histochemical and confocal laser scanning microscopy study of the bone-titanium interface: An experimental study in rabbits. Biomaterials 1994;15(3):194–200.
- Piattelli A, Scarano A, Piattelli M. Detection of alkaline and acid phosphatases around titanium implants: A light microscopical and histochemical study in rabbits. Biomaterials 1995;16(17):1333–1338.
- Garetto LP, Chen J, Parr JA, Roberts WE. Remodeling dynamics of bone supporting rigidly fixed titanium implants: A histomorphometric comparison in four species including humans. Implant Dent 1995;4(4):235–243.
- Steflik DE, Noel C, McBrayer C, Lake FT, Parr GR, Sisk AL, Hanes PJ. Histologic observations of bone remodeling adjacent to endosteal dental implants. J Oral Implantol 1995;21(2):96–106.
- Buser D, Ruskin J, Higginbottom F, Hardwick R, Dahlin C, Schenk RK. Osseointegration of titanium implants in bone regenerated in membrane-protected defects: A histologic study in the canine mandible. Int J Oral Maxillofac Implants 1995;10(6):666–681.