

Ex vivo surface and mechanical properties of coated orthodontic archwires

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SUMMARY This study examined the mechanical and physical properties of retrieved coated nickel–titanium (NiTi) archwires compared with unused samples. Ultraesthetic™ 0.016 inch coated archwires (G&H® Wire Company) were investigated. Ten as-received wires were subjected to a three-point bending test using conventional and self-ligating bracket systems. Surface roughness of the coating was measured with a contact stylus profilometer. Optical and scanning electron microscopes were used to assess surface topography. Ten archwires were used *in vivo* for a period of between 4 and 6 weeks. Retrieved archwires were subjected to the same tests. The percentage of the remaining coating was calculated using digital photography. Coated archwires were used *in vivo* for a mean period of 33 days. Differences between the mean values of the as-received and retrieved archwires were determined using *t*-tests.

In the three-point bending test, with conventional elastomeric ligation, retrieved wires produced a lower unloading force ($P < 0.001$). Both retrieved and as-received coated archwires produced zero values of unloading force when deflected for 4 mm. When tested using a self-ligating bracket system, retrieved and as-received coated archwires produced the same amount of force ($P > 0.05$). With surface profilometry, all measured roughness parameters (except R_{sm}) had greater surface roughness for the retrieved coated archwires ($P < 0.05$). Under microscopy, retrieved coated archwires showed discolouration, ditching, and delamination. Only 75 per cent of the coating was present in retrieved coated archwires.

Retrieved coated archwires produced lower unloading force values than as-received coated archwires with conventional ligation. Surface roughness of coated archwires increased after use. Coated archwires have a low aesthetic value, with 25 per cent of the coating lost within 33 days *in vivo*.

Introduction

There is a growing demand for aesthetic appliances, yet most fixed orthodontic appliance components are metallic and silver in colour. This problem was partially solved by the introduction of aesthetic transparent brackets made of ceramic or composite (Russell, 2005). However, archwires are still made of metals such as stainless steel and nickel titanium (NiTi). Coating metallic archwires with plastic resin materials is currently the only existing solution to this aesthetic problem. Materials used in the coating process are Teflon® or epoxy resin. The process of applying this layer includes using clean compressed air as a transport medium for the atomized Teflon® particles to coat the wire which is further heat treated in a chamber furnace (Husmann *et al.*, 2002).

There are different opinions in the literature concerning coated archwires. Husmann *et al.* (2002) evaluated their sliding properties and the adherence of the coating to the archwires. They found that plastic coating decreased friction between archwires and brackets. On the contrary, Proffit (2000) described this coat as ‘undurable’. Other authors have experienced some difficulties with these coated archwires, claiming that the colour tends to change with time and that the coating splits during use in the mouth, exposing the underlying metal (Postlethwaite, 1992; Lim

et al., 1994; Kusy, 1997). No studies have been conducted to examine the mechanical properties and to quantify the amount of coating that remains after the use of these wires in the oral cavity.

Mechanical properties of orthodontic archwires can be assessed using a three-point bending test. This evaluates the load-deflection properties, which are considered the most important parameters determining the biological nature of tooth movement (Krishnan and Kumar, 2004) and provides information on the behaviour of the wires when subjected to deflection in the horizontal and vertical directions (Kapila and Sachdeva, 1989). The advantages of this test are the close simulation to clinical application and its ability to differentiate wires with superelastic properties (Krishnan and Kumar, 2004). In addition, it offers a high degree of reproducibility which facilitates comparison between different studies (Wilkinson *et al.*, 2002). The wire is deflected and the load generated inside is measured. The load-deflection curve generated is analysed to detect the mechanical properties of the wires.

Surface roughness of orthodontic archwires is an essential factor in determining the effectiveness of archwire-guided tooth movement (Bourauel *et al.*, 1998). The surface quality of archwires also affects the area of surface contact and influences its corrosion behaviour and biocompatibility. In

addition, it has a role in colour stability and performance of the appliance using sliding mechanics (Kusy *et al.*, 1988; Bourauel *et al.*, 1998). However, Kusy and Whitley (1990) found that although the surface roughness of different wires showed positive correlation with their coefficient of friction, frictional loss and the rate of orthodontic tooth movement is a more complex multifactorial process. There are several methods used to measure surface roughness of orthodontic archwires, such as contact surface profilometry, atomic force microscopy, and laser spectroscopy. Bourauel *et al.* (1998) compared the surface roughness of different wires using these three techniques. They found that, in most cases, there was high correspondence between all three methods. The contact stylus tracing method, known as surface profilometry, is probably the most commonly used technique to study the surface roughness of materials. Three types of parameters are used to describe surface topography: amplitude (R_a , R_t , and R_q), spacing (R_{sm}), and hybrid (Rd_q). In order to have a clear image of surface roughness of a certain material, each of these parameters should be represented in the results.

The aims of this research were to investigate the mechanical properties (three-point bending test), surface topography (surface roughness), and surface coating (optical characterization) of coated superelastic archwires before and after clinical use.

Material and methods

The retrieved, *ex vivo*, archwires were obtained from subjects in a randomized clinical trial studying the efficiency of these coated archwires in levelling and alignment of teeth. Ethical approval was obtained for this investigation from the Salford and Trafford Local Research Ethics Committee (Ref: 05/Q1404/139). These were new patients seeking treatment that met the following criteria:

1. Upper and lower fixed appliance treatment planned.
2. No anterior tooth extraction or proximal stripping during treatment.
3. No congenitally absent anterior teeth.
4. Pre-treatment Little's Irregularity Index >5.0 mm.
5. No congenital clefts or craniofacial syndromes.

The patients were randomly allocated to either conventional or epoxy resin-coated NiTi archwires. Orthos® brackets (Ormco, Orange, California, USA) with a slot size of 0.022×0.028 inch were bonded. A 0.016 inch archwire, either Ultraesthetic™ or NiTi (G&H® Wire Company, Greenwood, Indiana, USA) was used as the first archwire placed to achieve initial alignment. The archwires were ligated into brackets using elastomeric modules. After 4 to 6 weeks, the archwires were retrieved and changed according to treatment progress. The operators were instructed to place the retrieved archwires into a prepared self-sealed envelope. On the outer surface of each envelope the date of placement,

date of retrieval, and the size and type of wire was recorded. For the purpose of this study, 10 retrieved 0.016 inch coated archwires were selected.

The procedures described by Eliades *et al.* (2000) for collecting retrieved archwires were followed. The wires were cleaned with distilled water to remove any precipitations. Specimens with a length of 20 mm were then prepared from the straight section of the archwires using wire cutters. The specimens were subjected to different tests.

For three-point bending, a jig with two parallel brass rods with a diameter of 5 mm was constructed. Two Orthos® edgewise central incisor brackets with a slot size of 0.022×0.028 inches were bonded on the top of these rods with an interbracket distance of 14 mm, equivalent to the distance between the central incisor and canine bracket. The jig was fixed to the base of Zwick/Roell Z020 universal testing machine (Zwick GmbH & Co. KG., Ulm, Germany). The wire specimen was attached to the brackets using elastomeric ligatures (Ormco, moulded 'O' 0.120 inches) in a standard configuration. Two self-ligating Damon 2® brackets (Ormco) were bonded at the same interbracket distance. The force was applied using a third brass rod attached to the moving part of the Zwick machine applied vertically toward the middle of the interbracket distance at a crosshead speed of 1 mm/minute for both loading and unloading. Testing was performed at room temperature as the archwires were not heat-activated NiTi. The mid portion of the wire was deflected first by 2 mm then unloaded, and secondly by 4 mm then unloaded. The loading and unloading forces were registered by the load cell at the rate of 100 measurements per second. The results were analysed with testXpert® software (Zwick GmbH & Co. KG) and unloading forces were reported at 2, 1.5, and 1 mm when wires were deflected for 2 mm and at 4, 3, 2, and 1 mm when deflected for 4 mm. The large number of measurements recorded resulted in measurement noise and the raw data graphs obtained were smoothed in the SigmaPlot Graphics software (Systat Software Inc., San Jose, California, USA) where the final curves were plotted. No specific algorithm was used.

A contact stylus profilometer (Taylor Hobson Precision Instrument, Taylor Hobson® Ltd, Leicester, UK) was used to measure the surface roughness of the archwires. This machine has a diamond stylus attached to the instrument arm. When the stylus is drawn across the surface by the drive unit, the tip accurately follows the profile of the surface by moving vertically up and down. The diamond stylus has a radius of 5 µm and a tip angle of 90 degrees and traverses at a constant speed of 1 mm/second across the surface with a force of 6 mN. Three line scans were performed per specimen surface. The cut-off length was 0.25 mm and the measuring length 1 mm. Each wire specimen was scanned at three different areas. Through an inductive transducer coupled to the stylus, data from each

trace was relayed to the central processing unit. Data was then translated into digital values for each surface parameter selected, using the software Ultra version 4.3.14. (Taylor Hobson® Ltd). Five different parameters were selected: R_a , R_q , R_t , R_{sm} , and Rd_q . The results were then exported to the Statistical Package for Social Sciences (version 15 for Windows, SPSS Inc., Chicago, Illinois, USA) which was used for statistical analysis of the results.

Optical and scanning electron microscopy (SEM) were used to assess the micromorphological characteristics of the archwires before and after use in the oral cavity. A 20 mm long specimen of each wire was mounted on a holder and observed under field-emission SEM (Philips XL 30, Eidhoven, The Netherlands). The images were recorded at $\times 100$ magnification.

A Meiji® light microscope (Meiji Technology Ltd, Tokyo, Japan) was used to study surface topography of the archwires at $\times 45$ magnification. The objective was to assess the surface topography of coated archwires and determine the presence of any cracks or penetrations along their surfaces.

Digital photographs of each side of each archwire were taken on a grey background using a HP Photosmart® 945 digital camera (Hewlett-Packard, Palo Alto, California, USA). The camera was fixed on a tripod and orientated at 90 degrees to the surface on which the archwire was placed. The camera to wire distance remained constant and a ruler was placed adjacent to the archwire for calibration purposes. Images were recorded of each side of the archwire, that is the gingival and occlusal aspects. Digital photographs were transferred to a computer and the Image J® program (W. Rasband, National Institute of Health, Bethesda, Maryland, USA) was used for analysis. The program was first calibrated

using the ruler in the photograph. The overall length of the wire and finally the length of the remaining coated segments were measured. The measurements were exported to Excel® (Microsoft Corporation, Redmond, Washington, USA). The percentage of the remaining coating was calculated by dividing the sum of the length of the remaining coated segments over the overall length of the wire. The average percentage of the two sides of each wire was then calculated.

A sample size calculation was undertaken using the data from mechanical testing by Walker *et al.* (2005). To detect a 10 per cent difference between groups with power of 0.80, a sample size of 10 wires per group was required.

Data were transferred from the testXpert® software to SPSS 15.0 for Windows. The data for subgroups were tested for normality, and then, *t*-tests were used to compare the means between groups. Only those including samples recording zero force on unloading showed significant deviation from normal distribution. No statistical tests were performed for these groups.

Results

Three-point bending test

Table 1 and Figure 1 show the results for the three-point bending tests using the different methods of ligation at the various loading and unloading stages.

Using conventional ligation in the three-point bending test, retrieved coated archwires generated lower unloading forces (15–29 g) when deflected for 2 mm compared with as-received coated archwires (46–59 g) which was statistically significant ($P < 0.001$). Figure 1a shows the clear differences in unloading behaviour. However, both retrieved and as-received coated archwires produced zero

Table 1 Means and standard deviations (SD) of force (in grams), for 0.016 inch Ultraesthetic™-coated archwires (before and after use) using elastomeric and self-ligation, under various forces of loading and types of deflection.

Amount and type of deflection	Initial deflection	Ligation type	Force (SD)		<i>P</i> value (<i>t</i> -test)
			As-received specimens	Retrieved specimens	
Loading : 2 mm deflection	2 mm	Elastomeric	192 (20)	201 (18)	0.427
Unloading: 1.5 mm deflection			59 (12)	29 (21)	<0.001
Unloading: 1.0 mm deflection			46 (9)	15 (15)	<0.001
Loading : 4 mm deflection	4 mm		320 (18)	326 (54)	0.725
Unloading: 3.0 mm deflection			0.0	0.0	
Unloading: 2.0 mm deflection			0.0	0.0	
Unloading: 1.0 mm deflection	2 mm	Self-ligation	0.0	0.0	
Loading : 2 mm deflection			157 (12)	149 (26)	0.436
Unloading: 1.5 mm deflection			61 (23)	65 (14)	0.745
Unloading: 1.0 mm deflection	4 mm		48 (12)	40 (17)	0.367
Loading : 4 mm deflection			217 (21)	203 (38)	0.328
Unloading: 3.0 mm deflection			53 (12)	38 (17)	0.071
Unloading: 2.0 mm deflection			51 (14)	41 (17)	0.288
Unloading: 1.0 mm deflection			38 (18)	30 (17)	0.415

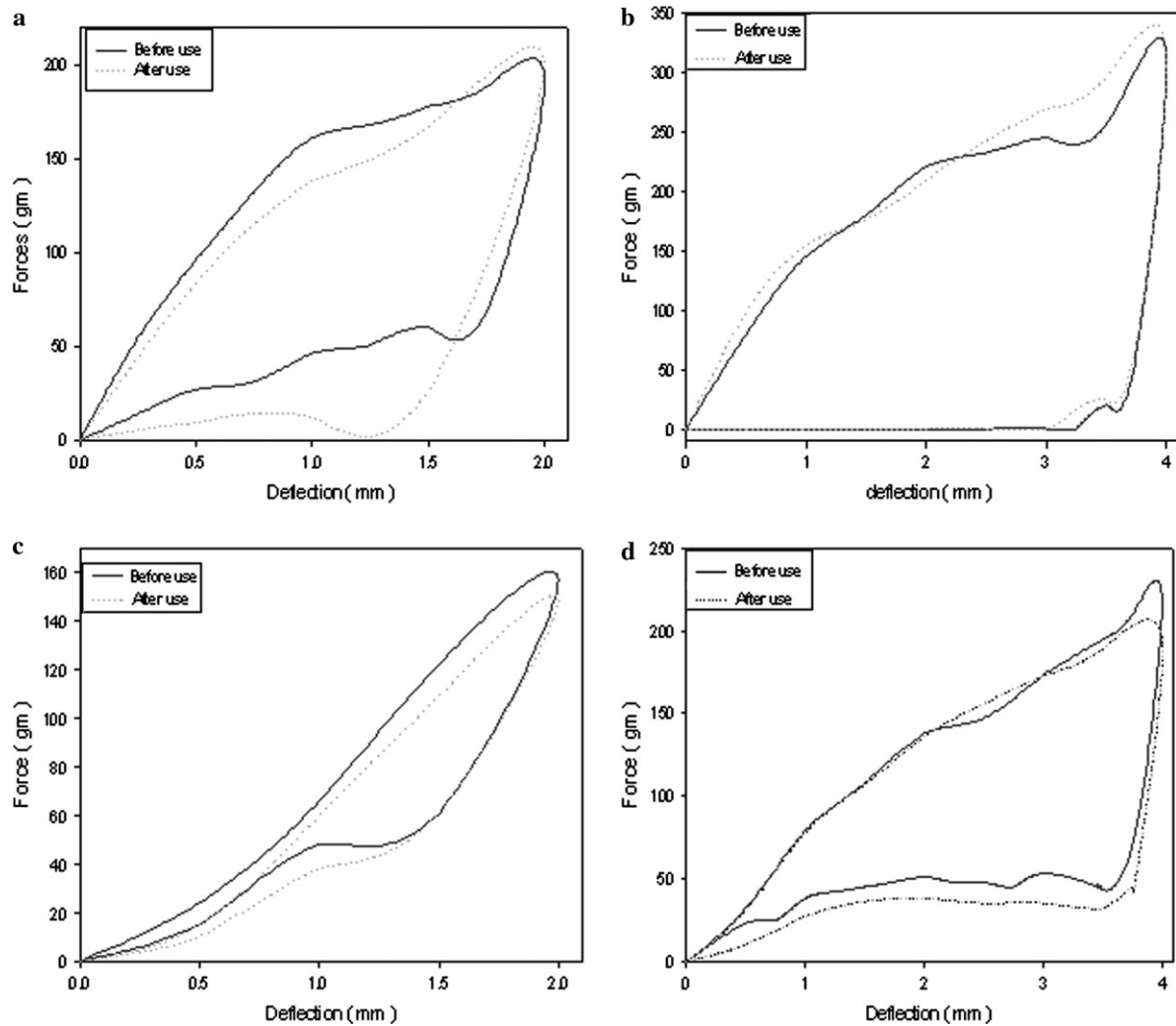


Figure 1 Representative force-deflection curves for 0.016 inch Ultraesthetic™-coated archwires before and after use *in vivo*: elastomeric ligatures and 2 mm deflection (a); elastomeric ligatures and 4 mm deflection (b); Damon2® self-ligating brackets and 2 mm deflection (c); Damon2® self-ligating brackets and 4 mm deflection (d).

unloading force when deflected for 4 mm. The similarity of the behaviour is seen in Figure 1b, along with the zero force delivery throughout most of the unloading phase. Using the Damon 2® self-ligating bracket system, retrieved and as-received coated archwires generated similar unloading force with both 2 mm (40–65 g) and 4 mm (30–53 g) initial deflection and there were no statistically significant differences. Figure 1c,d not only illustrates the similarities in the as-received and retrieved specimens but also the different unloading behaviour of the wire with the greater deflection where the plateau effect is clearly seen.

Surface roughness

Coated archwires demonstrated rougher surfaces after use *in vivo*. R_a , R_t , and Rd_q showed statistically significantly higher values ($P < 0.05$). R_a also showed a higher value,

Table 2 Mean values of roughness parameters of 0.16 inch Ultraesthetic™ coated archwires before and after use *in vivo* for a mean period of 33 days.

	Before use	After use	Paired sample <i>t</i> -test
R_a (μm)	0.92 (0.33)	1.25 (0.86)	0.056
R_q (μm)	1.16 (0.40)	1.86 (1.28)	0.006
R_t (μm)	6.59 (2.24)	13.43 (10.00)	0.001
R_{sm} (μm)	186.4 (30.03)	221.3 (141.0)	0.195
Rd_q ($^\circ$)	3.05 (1.33)	6.58 (3.80)	0.00

although not statistically significant ($P = 0.056$). The spacing parameter, R_{sm} , also increased but was not statistically significant ($P = 0.195$). Comparison of surface roughness parameters for as-received and retrieved 0.016 inch coated archwires are shown in Table 2.

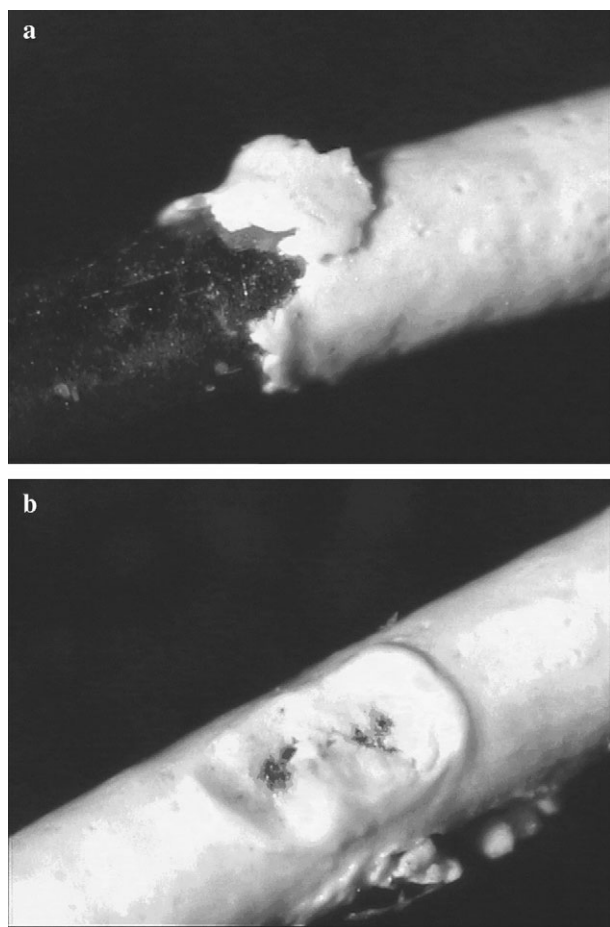


Figure 2 Reflected light images of retrieved 0.016 inch coated archwire at a magnification of $\times 45$. Coating peeled off the archwire (a) and an imprint of a bracket (b).

Surface topography

Figure 2a,b shows reflected light images and Figure 3a,b the scanning electron micrographs of retrieved 0.016 inch coated archwires. Delamination of the coating over large areas was obvious. In most cases, the coated archwires showed discolouration, ditching, and cracking. Images revealed that the coating showed high and variable amounts of deterioration.

Quantitative analysis demonstrated that, on average, 25 per cent of the coating was lost over the length of the retrieved archwire, exposing the metallic surface below.

Discussion

This study has identified important changes in the properties of aesthetic coated archwires after clinical use. This *ex vivo* or 'retrieval analysis' methodology has been used for a long time in orthopaedic research and has recently gained interest in dental materials investigations (Eliades *et al.*, 2000). It provides critical information concerning the performance of materials in the environment in which they are intended to

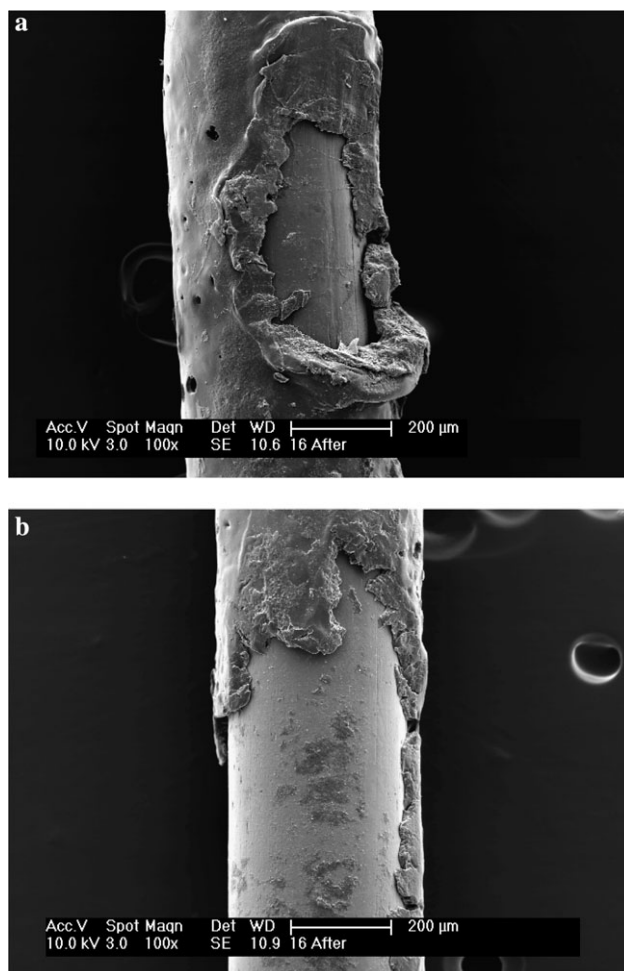


Figure 3 Scanning electron micrographs showing the deterioration of the coating of the retrieved 0.016 inch coated archwires at a magnification of $\times 100$. Part of the coating is missing (a) and delamination of the coating (b).

operate. In addition, this method helps in the assessment of alterations of material properties that can occur during clinical use. However, it is not able to describe the sequence in which the alterations occur.

Mechanical properties

Retrieved 0.016 inch coated archwires tested with conventional elastomeric ligation generated a lower unloading force ($P < 0.001$) compared with as-received coated archwires at 1.0 and 1.5 mm deflection. This is most likely due to increased frictional forces between the irregular surface of the coating and brackets. The test set-up was chosen from the many available in the literature as one that mimics the complex situation encountered *in vivo* (Miura *et al.*, 1986), where the effects of friction and binding are overlaid on the effect of wire deflection. This increased friction reduces unloading and increases loading forces. Retrieved and as-received aesthetic archwires both showed zero unloading force when deflected for 4 mm using elastomeric ligation. Examination of these samples

showed that the wires were unable to slide back through the ligated brackets and the archwire remained static as the load was released. This is due to excessive friction between the archwire and the bracket and is most likely due to damage to the coating 'jamming' the wire in place. This effect of the bracket edges digging into the archwire coating was also observed in the retrieved samples when the surface topography was examined and bracket imprints could be visualized. This indicates that the efficiency of coated archwires to generate force to produce tooth movement may be considerably reduced when used with conventional ligation in circumstances where the archwire undergoes large deflections.

With the Damon2® passive self-ligating bracket system, retrieved 0.016 inch coated orthodontic archwires showed no statistically significant difference in loading and unloading force ($P > 0.05$) compared with the as-received coated archwires at various loading and unloading deflections. This appears to be due to the fact that passive self-ligating brackets produce low friction and are not affected by the increased surface roughness and deterioration of the coating in the retrieved samples. These findings demonstrate the benefits of using passive self-ligating brackets with coated archwires and indicate that the findings with elastomeric ligation are due to frictional effects.

Surface roughness

The parameters, R_q , R_t , and Rd_q showed statistically higher values, indicating greater surface roughness for the retrieved coated archwires. These results show that the heights of valleys and peaks of the surface increased while the number of these peaks and valleys per unit measurement did not increase to the same extent. The use of different types of surface roughness parameters is crucial in analysing the results. This finding is in agreement with those of Wichelhaus *et al.* (2005). They reported an increase in surface roughness of different wires after 4 weeks of use in the mouth. This could be explained by the abrasive influence of tooth brushing and the interaction between the archwire coating and bracket edges.

Surface topography

Examination of the retrieved coated archwires using light microscopy showed interesting results. The coating suffered from high and variable amounts of deterioration. Many of the specimens were characterized by delamination of the coating over large areas. This clearly affects the aesthetic value of coated archwires.

The coating remained intact in some areas; however, it showed a rougher discoloured and deteriorated surface when compared with as-received archwires. These findings are in agreement with the increase in surface roughness parameters of retrieved coated archwires observed in the present study.

It was noted that imprints of brackets and areas of delamination were related to the positions of the brackets in most samples. This finding in the retrieved wires may further explain the increase in friction found during mechanical testing, as such surface defects related to the edges of the brackets occurring during the three-point bending test would impede the archwire sliding through the bracket.

On average 25 per cent of the coating was lost during use which led to significant reduction of the aesthetic qualities. The coating peeled off in many areas during *in vivo* use leaving surface defects. The irregular surfaces found microscopically may lead to plaque accumulation in surface defects and tooth movement may be affected due to entrapment of bracket edges inside these defects. This agrees with the opinions of others (Postlethwaite, 1992; Kusy, 1997; Proffit, 2000; Neumann *et al.*, 2002).

Quantitative measurements of the amount of lost coating have not been previously reported. The method developed for this can be described as simple, valid, and reproducible. This method was found to be very accurate, as demonstrated by comparing the length of the two sides of each wire where the largest difference was 0.1 mm, a 1 per cent difference.

Conclusions

1. Retrieved coated archwires produce lower unloading force values than as-received coated archwires with conventional ligation, and the same force values with self-ligating brackets.
2. Surface roughness of coated archwires increases after use *in vivo*.
3. Coated archwires had a low aesthetic value. Twenty five per cent of the coating was lost within 33 days *in vivo* and surface morphology showed severe deterioration.

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