ORIGINAL ARTICLE

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In-vitro evaluation of the corrosion behavior of orthodontic brackets

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Structured Abstract

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Objective – The number of bracket systems for orthodontic therapy increases significantly. One major concern of newly developed orthodontic devices is aspects of corrosion and biocompatibility.

Material and Methods – In this study, nine bracket systems made of different material and from various design principles were tested with respect to their corrosion behavior. Electrochemical and static immersion tests with subsequent measurement of nickel ion release were performed. In addition surface alterations of the brackets after corrosion were documented by scanning electron microscopy. Studies of corrosion behavior were performed according to the DIN/ISO standard 10271 for corrosion testing of dental materials.

Results – All systems showed traces of corrosion after electrochemical testing. However, after static immersion testing only minor corrosion defects could be documented and the measured nickel ion release was far below critical limits.

Conclusions – All tested systems seem to be biocompatible and applicable for orthodontic therapy. The measured nickel values are far below the daily dietary intake level. A static immersion test combined with the nickel ion release measurement seems to be more relevant for the determination of biocompatibility than the electrochemical testing.

Key words: brackets; corrosion; electrochemical test; nickel release; orthodontics

Introduction

Today most of the brackets used for orthodontic therapy are made of alloys (1–5). These materials can easily be formed by casting and milling. A new method of bracket production is the metal injection molding (MIM) technique, getting more and more important in the last years (1). Some other brackets are built by fiber-reinforced composite or polycarbonate (6). Independent of the production process all metallic appliances are affected by corrosion (7, 8). Corrosion is an electrochemical reaction of a metal or alloy with different components of its environment (9). Brackets are subject to corrosion in the oral cavity because they are immersed in the patient's saliva, acting as an electrolyte. Additional factors influencing corrosion are varying oral temperatures, the presence of plaque and the daily dietary intake (7). Oxygen required for corrosion is present in abundance.

Two major aspects of corrosion are of importance: First of all, corrosive processes result in the destruction of the surface by a loss of metal ions. In general, surface corrosion acting on the whole metal surface is considered to be less destructive than local corrosion effects, as for example pitting corrosion. Localized corrosive attacks can weaken the structure and result in fracture. Secondly, the problem of ion release into the oral cavity is discussed because of its biological effects. The recent literature shows the wide attention in attributes of dental alloys such as cytotoxicity and allergenicity (7, 10–12).

The purpose of this study was to determine corrosive processes on classical and self-ligating orthodontic brackets by a static immersion test combined with the analysis of the nickel ion release into the environmental solution and a potentio-dynamic electrochemical test. Furthermore, the surface structures were to be evaluated by scanning electron microscopy (SEM) before and after electrochemical exposure to show the surface changes as a result for the bracket itself. The nickel ion release into artificial saliva was measured by inductively coupled mass spectrometry (ICP-MS). Using this technique a quantitative value for the assessment of the biocompatibility of the brackets under investigation could be determined.

Material and methods

Material

Nine commercially available types of brackets were included in this study (Table 1). The systems chosen for this study differed in design, material and functional aspects. All systems but Discovery® (Dentaurum, Ispringen, Germany) and Ultratrimm® (Dentaurum, Ispringen, Germany) were self-ligating brackets. The Damon® 3 (Ormco Corporation, Orange, CA, USA) bracket is assembled of a special composite resin body and metallic slot and passive clip. For the purpose of comparison, the behavior of Damon® 2 (Ormco Corporation, Orange, CA, USA) can be taken as evaluation criterion, as the metallic part can be assumed to be manufactured from the same material and the body of the two brackets are roughly of the same dimension. The metal brackets could be divided into brackets produced by MIM (Opal™-M [UP Dental GmbH, Köln, Germany], Discovery®) and milled brackets (SPEED™ [Strite Industries Limited, Cambridge, Ontario, Canada], Damon® 2, Smartclip™ [3M Unitek, St. Paul, MN, USA], In-Ovation® [Dentsply GAC International Inc., Bohemia, NY, USA], TIME® [AD adenta GmbH, Gilching, Germany], Ultratrimm®).

Table 1. **Types of brackets tested in this study**

Bracket	Design and material	Manufacturer
Damon® 2	Steel, MIM clip, self-ligating	Ormco
Damon® 3	Composite, steel clip and slot, self-ligating	Ormco
Discovery®	MIM, conventional clip	Dentaurum
In-Ovation®	Milled steel, self-ligating	Dentsply GAC
Opal™-M	MIM, self-ligating	UP Dental
SPEED™	Milled steel, self-ligating, active NiTi clip	Strite
SmartclipTM	Milled steel, NiTi clip, self-ligating	3M Unitek
TIME®	Milled steel, self-ligating	AD adenta
Ultratrimm®	Milled steel, conventional clip	Dentaurum

Static immersion tests and measurement of Ni ion release

The static immersion test was performed on the basis of the DIN/ISO standard 10271 (13). Ten test tubes of borosilicate glass with a volume of 10 ml each were used. A single bracket was placed into each vessel. After cleansing with alcohol the brackets were stored in 10 ml of artificial saliva with a modified Fusayama composition (Table 2, 14). Artificial Fusayama saliva was used instead of a lactic acid-based solution in order to better simulate oral conditions as described in Annex A, A.3.3 and A.5 of the standard (13). Prior to storage of the brackets in the solution its pH value was measured. If the pH value was in a range of 4.8–5.0 the solution was filled into the glass containers, otherwise

Table 2. **Composition of the artificial Fusayama saliva used in this study (14)**

Components	(mg/l)
Sodium chloride	400
Potassium chloride	400
Calcium chloride-dihydrate	795
Sodium hydrogen phosphate-1-hydrate	690
Potassium rhodanide	300
Sodium sulfide	5
Urea	1000

it was refused and a new solution was prepared. A temperature control circuit with a constant value of 37°C adjusted the temperature of the solution. After an immersion period of 1 week the brackets were removed from the solution. The test solution of each bracket was stored in closed glass vessels until nickel ion measurement. The artificial saliva was analyzed and the nickel ion concentration was measured using an inductively coupled plasma mass spectrometer (ICP-MS ELAN 5000, Perkin Elmer, Wellesley, MA, USA).

Electrochemical tests and determination of breakdown potentials

The electrochemical testing was performed in accordance to the DIN/ISO standard 10271 (13, 15). An electrochemical cell was constructed using an Ag/AgCl reference electrode (Type B 2820, Schott, Mainz, Germany), a platinum counter electrode, a Haber-Luggin capillary (salt bridge), a gas inlet/outlet for N_2 rinsing and a temperature controlling system (see Fig. 1). The nitrogen was needed to replace the oxygen to prevent unfavorable reaction between the oxygen and the electrodes. The electrochemical cell was driven by a computer-controlled scanning potentiostat (MLab SCI, Bank Electronic-Intelligent Controls GmbH, Clausthal-Zellerfeld, Germany) with a potential range ± 1600 mV. There were only three modifications as compared with the setup defined in the standard DIN/ISO 10271: (1) The cell had a temperature of 37°C to simulate body temperature. (2) Modified Fusayama saliva was used to simulate the *in vivo* conditions in the oral cavity. (3) The brackets used as specimens had an undefined surface not equal to the 1 cm^2 as defined by the DIN/ISO 10271. All measured potentials were converted to saturated calomel electrode values to be compatible. Ten brackets of each system were used as specimens. Each specimen was exposed to the artificial saliva as a working electrode and potentials were measured as defined by the standard.

Experimental procedure to determine breakdown potentials

Ten specimens of each system were tested. At first the artificial saliva was mixed as electrolyte and the pH measured before filling up the borosilicate vessel. The temperature was adjusted to 37°C prior to the start of the experiment. Simultaneously nitrogen was slightly blown in for more than 30 min into the solution to desorb the oxygen. The brackets were fixed onto a titanium molybdenum wire using orthodontic elastics and placed into the solution. The titanium molybdenum wire was chosen because of its extremely high corrosion resistance. The breakdown potential of that wire was determined to be above 2000 mV and thus was far beyond any other breakdown potential of dental materials determined in other studies (15). The surface area that was in contact with the bracket and the solution was minimized by choosing a round wire and by placing the bracket as near to the solution surface as possible in order not to falsify the electrochemical results too much. The open circuit potential was measured for a period of

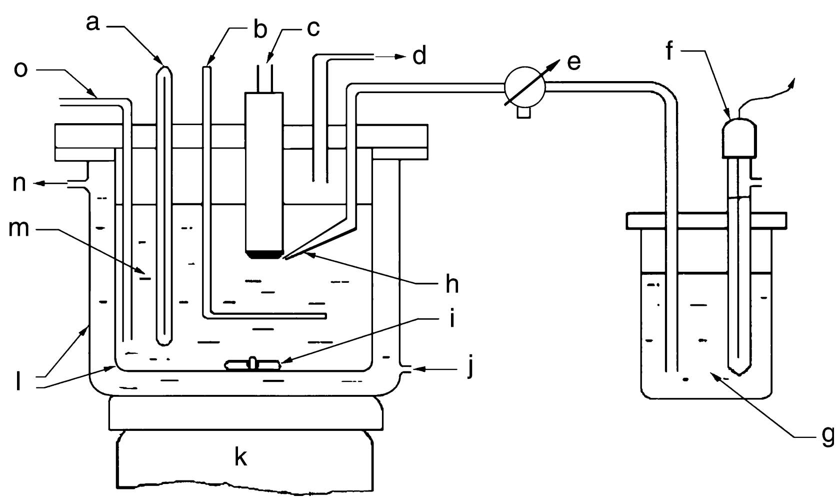

Fig. 1. Schematic diagram of the electrochemical cell (13): (a) Thermometer, (b) Counter electrode, (c) Working electrode, (d) Gas outlet, (e) Electrolytic bridge, (f) Reference electrode (saturated calomel electrode), (g) Saturated solution of KCl, (h) Lugging capillary, (i) Magnetic stirrer (PTFE-coated), (j) Water inlet, (k) Magnetic agitator (motor), (l) Double-walled borosilicate vessel, (m) Electrolyte, (n) Water outlet, (o) Bubbler (using nitrogen).

2 h. According to ISO 10271, the starting point for polarization was set to 150 mV below the value of the open circuit potential, and the potentio-dynamic scan was performed up to a potential of 300 mV above the breakdown potential with a sweep rate of 1 mV/s. The breakdown potential was extracted from the measured current density/potential curve.

Investigation of surface changes by SEM

Scanning electron microscopy (SEM, Philips XL 30, FEI Company, Eindhoven, The Netherlands) was used to analyze the bracket surfaces prior to and after exposure to the electrochemical test. The brackets were examined in the as-received and the corrosion state in order to be able to analyze the changes caused by corrosion and to see which structures were the results of the production processes. The brackets were cleansed with 90% ethanol. The composite component of the Damon® 3 bracket was sputter coated prior to the SEM inspection using a Scancoat six® sputter device (Edwards, Crawley, Great Britain) and a gold-platinum target. Each bracket was photographed as overview and detailed micrograph from each side of the bracket. Standard magnifications were set to 200×, 500× and 1,000×. In case there was a doubt whether corrosion processes or surface contamination was detected, the composition of the substance was analyzed using Energy Dispersive X-ray Spectroscopy (EDX, Genesis 4000, EDAX AMETEK GmbH, Taunusstein, Germany) to differentiate these two possibilities.

Results

Static immersion tests/Ni ion release measurement

The nickel ion release of the brackets measured with the ICP-MS did analyze the concentration of the nickel isotope 60-Ni in the corrosion media. Basically, 58-Ni has a higher natural abundance, but it coincides with 58-Fe, which aggravates data analysis. The values measured with the ICP-MS represented the Ni release of 1 week. Values were thus converted into nickel ion release per bracket per day.

The nickel ion release in single measurements ranged from a minimum of 0.01 μg/day for one TIME® bracket to a maximum of 5.24 μg/day (Discovery® bracket). The averages are shown in Table 3. The medians determined for 10 brackets of a brand can be divided into three groups: (1). <0.1 μg/day for the Smartclip™ system, (2). from 0.1 to 1.0 μg/day for Damon® 3, OpalTM-M, SPEED™, TIME® and Ultratrimm® and (3). more than 1.0 μg/day for the Damon® 2, Discovery® and In-Ovation® bracket systems (see Fig. 2). Statistical significance was tested for all results performing a *t*-test. *t*-test values are presented in Table 4, most values displayed significant differences.

Table 3. **Median values of the nickel ion release per bracket (μg/day) the breakdown potential and the open-circuit potential (mV)**

Bracket system	Nickel ion release	Breakdown potential	Open-circuit potential
Damon® 2	1.31	368	63
Damon® 3	0.41	233	100
Discovery®	1.82	884	90
In-Ovation®	1.47	480	34
Opal™-M	0.30	1317	88
Smartclip™	0.06	785	59
SPEED™	0.38	471	65
TIME®	0.23	365	78
Ultratrimm®	0.98	110	150

Electrochemical tests/open-circuit and breakdown potential

The breakdown potentials of all systems were within the working range of the test setup (±2000 mV). The results can be grouped in four categories. The lowest values were measured for the brackets of the Ultratrimm® system with 110 mV and for the Damon® 3 system with 233 mV (see median values in Table 3). The lower values ranged from about 365 to 480 mV (TIME®, Damon® 2, SPEED™, In-Ovation®). Higher values were measured for the brackets of the Discovery® (884 mV) and the Smartclip™ systems (785 mV). The highest breakdown potential was determined for the Opal™-M bracket with a breakdown potential of 1317 mV (Fig. 3). A *t*-test was performed to test all results for statistically significant differences. As can be seen from Table 5, most values showed significant differences.

SEM analysis of the exposed bracket surfaces

The following exemplary scanning electron micrographs demonstrate the corrosive processes registered for the different systems after electrochemical testing.

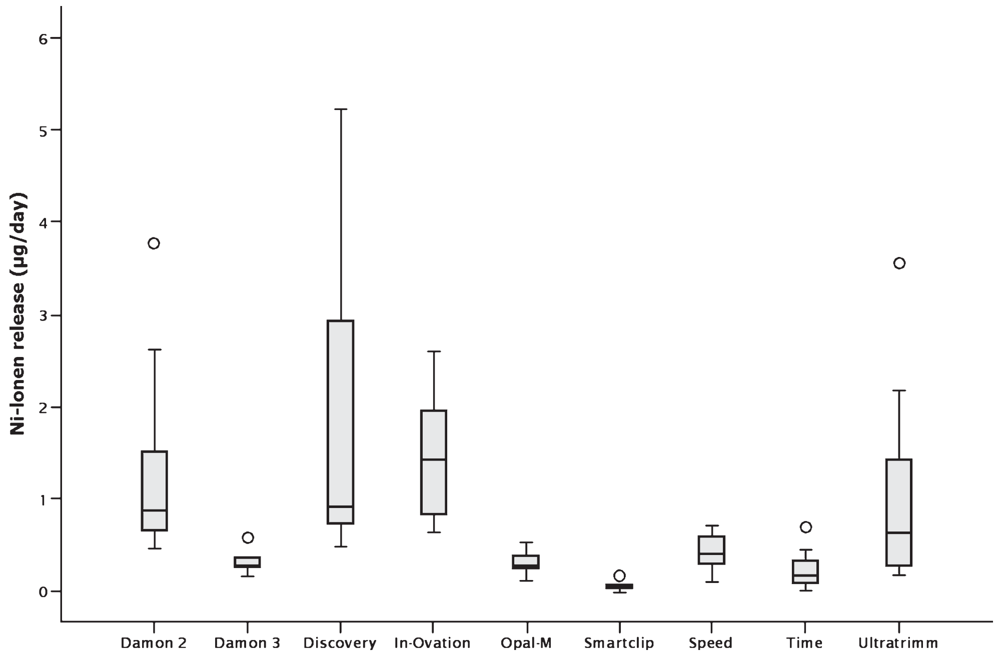

Fig. 2. Ni ion release in static immersion testing. The boxes with error bars represent 25 and 75% quartiles, the median, the SE and outliers (circles above bars).

Table 4. **Statistic analysis of the static immersion test results performed by the *t*-test**

	Damon® 3	Discovery	In-Ovation	Opal-M	Smartclip	SPEED	TIME	Ultratrimm
Damon 2	< 0.05	0.42	0.69	< 0.05	< 0.05	< 0.05	< 0.05	0.55
Damon 3		< 0.05	< 0.05	0.32	< 0.05	0.81	0.14	0.19
Discovery			0.54	< 0.05	< 0.05	< 0.05	< 0.05	0.87
In-Ovation				< 0.05	< 0.05	< 0.05	< 0.05	0.64
Opal-M					< 0.05	0.40	0.38	0.16
Smartclip						< 0.05	< 0.05	0.12
SPEED							0.16	0.18
TIME								0.15

The Damon® 2 bracket obviously showed signs of corrosion. Pitting corrosion can be found in Fig. 4A and is clarified in the detail of Fig. 4B. It appears at the head of the powerhook and at the base. The diameter of the larger defects is about 150–200 μm. The head of the hook shows a larger number of overlapping defects. The Damon® 3 brackets showed corrosive defects at the clip (Fig. 5A, B). The rest of the bracket showed no signs of corrosion because of its composite material. The Discovery® brackets predominantly showed a uniform distribution of corrosion. Only a few localized defects are found at the crossover part between bracket bases and the body (Fig. 6). The Opal™-M showed minor sign of uniform corrosion (Fig. 7), no localized pitting corrosion was visible. A large number of pitting corrosion defects could be observed for the Ultratrimm® brackets. The defects had diameters of about 100 μm (Fig. 8A). Most of these defects were detected at the bases of the brackets and at the hook (Fig. 8B).

Discussion

The results presented in this study have to be discussed with regard to the aspects of biocompatibility. The values measured were taken after static immersion testing of 1 week. Determination of the nickel release for 1 day requires division of the nickel ion release by seven, even though it can be expected that most nickel will be released during the first couple of days. The highest value of nickel release was 3.5 μg/day for the Discovery® system (Table 3). This value is almost two

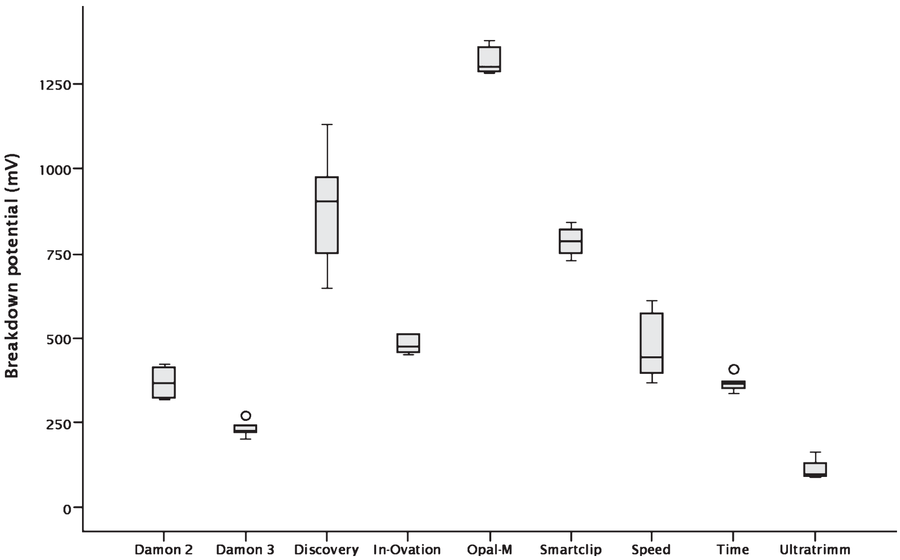

Fig. 3. Breakdown potential of the brackets.

Table 5. **Statistic analysis of the electrochemical test results performed by the *t*-test**

	Damon 3	Discovery	In-Ovation	Opal-M	Smartclip	SPEED	TIME	Ultratrimm
Damon 2	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	0.054	0.92	0.05
Damon 3		< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Discovery			< 0.05	< 0.05	0.22	< 0.05	< 0.05	< 0.05
In-Ovation				< 0.05	< 0.05	0.84	< 0.05	< 0.05
Opal-M					< 0.05	< 0.05	< 0.05	< 0.05
Smartclip						< 0.05	< 0.05	< 0.05
SPEED							< 0.05	< 0.05
TIME								< 0.05

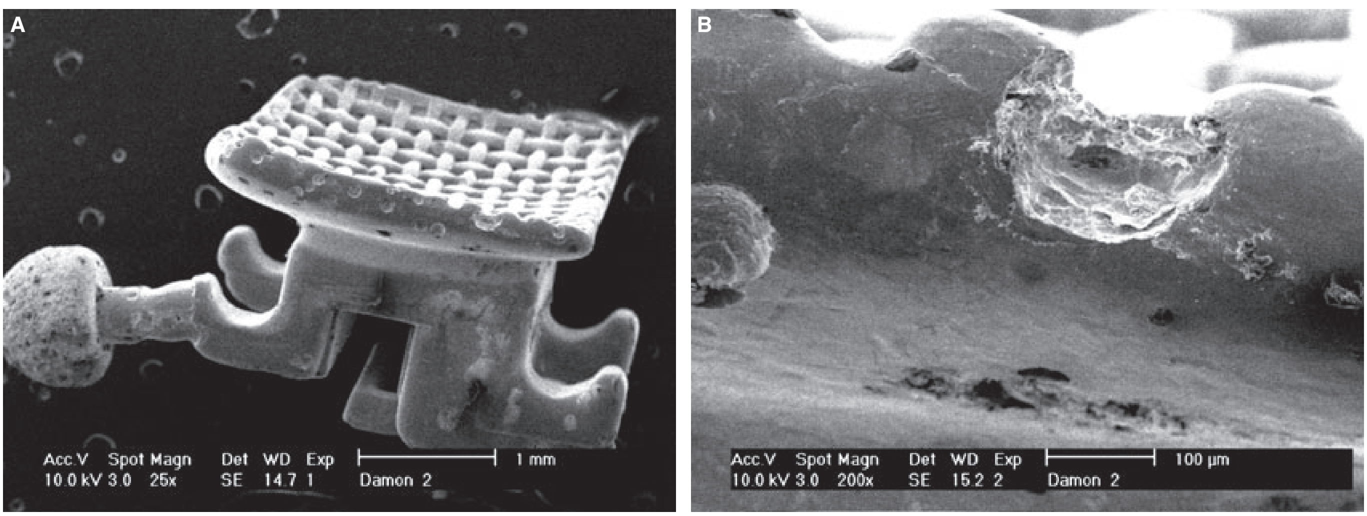

Fig. 4. (A) Damon® 2 bracket at 25× magnification. (B) Damon® 2 bracket detail at 200× magnification.

orders of magnitude below the daily dietary intake level (300–500 μg, 16) and the critical concentration (600–2500 μg) necessary to induce nickel allergy (17).

The results of the immersion test are in good agreement with recent literature (18, 19). The nickel ion release reported in these papers was in the same order

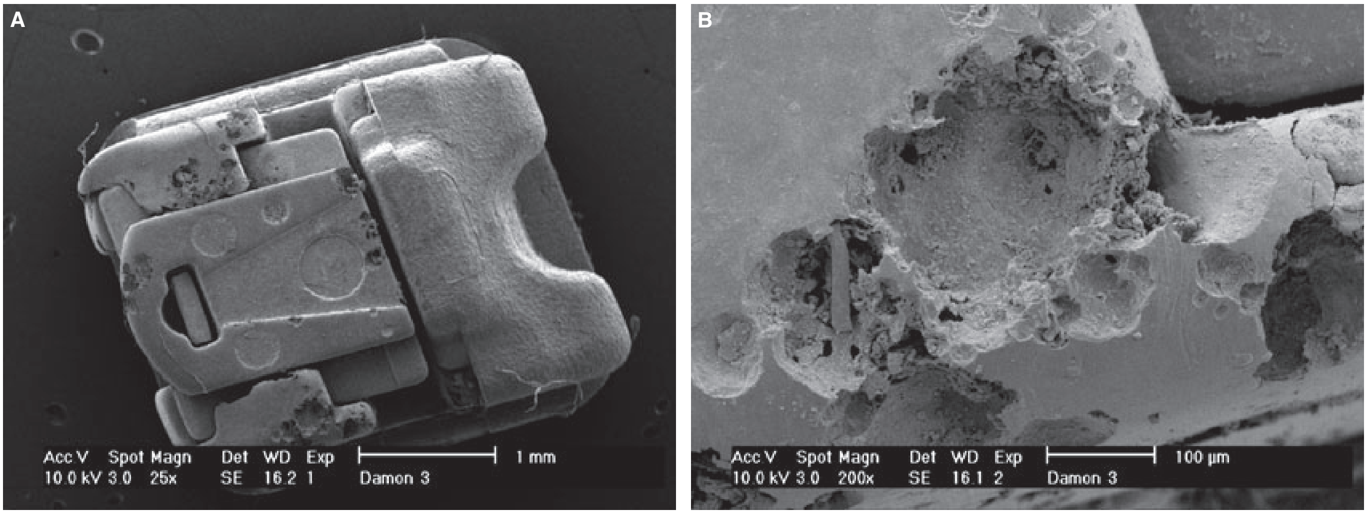

Fig. 5. (A) Damon® 3 bracket at 25× magnification. (B) Damon® 3 bracket detail at 200× magnification.

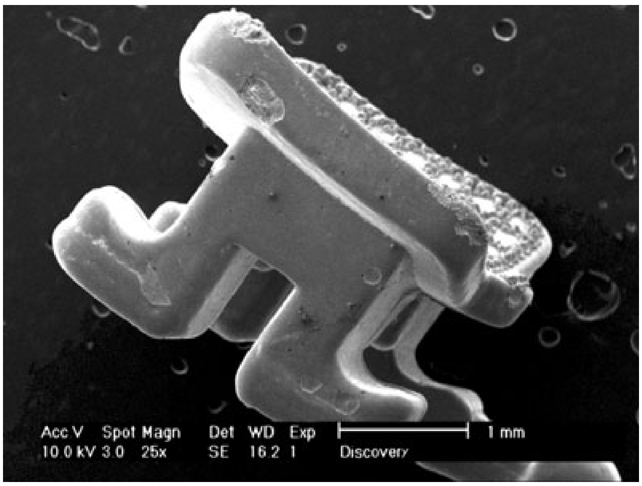

Fig. 6. Discovery® bracket at 25× magnification.

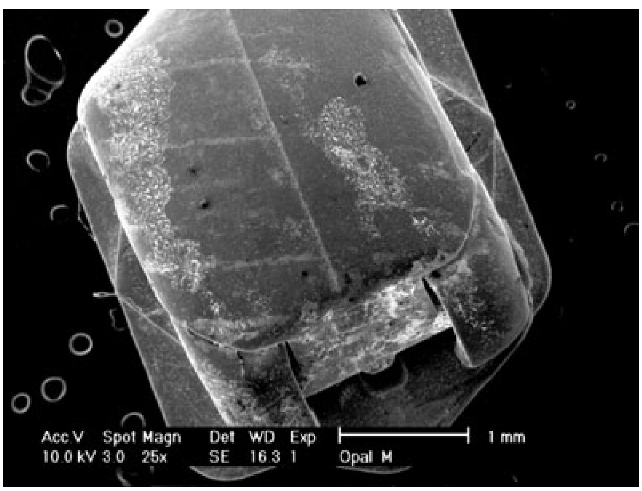

Fig. 7. Opal™-M bracket at 25× magnification.

of magnitude as in this study. Nevertheless it is important to notice that the test solutions applied differ from the Fusayama solution chosen in this study, which might explain the tendency towards higher Ni ion release in the presented study. While numerous earlier studies concentrated on the measurement of the breakdown potential, in the present study, both the corrosion behavior under electrochemical testing and the nickel ion release in static immersion tests of the brackets have been determined.

Measuring the release of metal ions should give some hints with regard to the clinical relevance of the previously determined electrochemical results. Obviously, a different and non-correlative behavior of the brackets in the two corrosion test methods must be noticed. Besides basic physical and electrochemical considerations that result in different behavior of the metallic materials in the two different corrosion tests, there are several further factors that have decisive influence on the breakdown potential on the one hand and the Ni ion release on the other hand. For example using a TMA wire to fix the brackets and immerse them into the solution for electrochemical testing might falsify the results as we now have a couple of the bracket material and the TMA wire in the corrosion solution. However, we assume that this is of minor influence, as the TMA wire has a very high breakdown potential and the influence should be nearly similar for all brackets tested. Furthermore, alloy composition and total surface area influence the behavior under electrochemical testing and the Ni ion release in immersion testing.

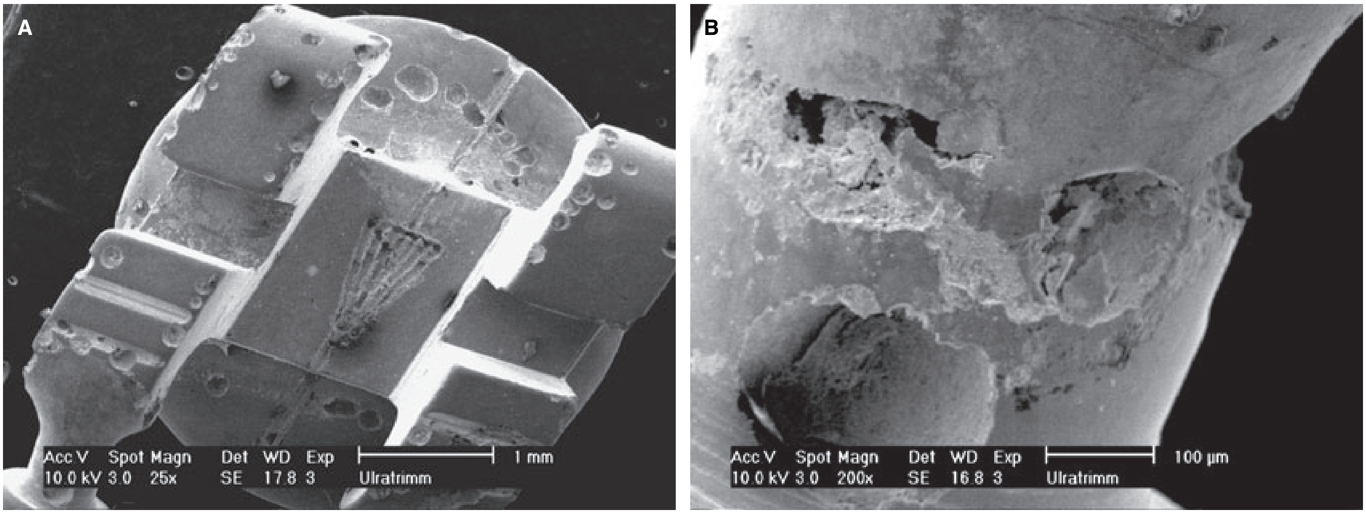

Fig. 8. (A) Ultratrimm® bracket at 25× magnification. (B) Ultratrimm® bracket, detailed view of the hook at 200× magnification.

However, these influences are hardly to quantify and the total surface area of a bracket is hardly to be determined exactly. Consequently, a direct correlation of electrochemical test results and Ni ion release under static immersion seems to be impossible, i.e. we feel that it is difficult to derive the intraoral corrosion behavior and biocompatibility from the electrochemical measurement of breakdown potentials. The following results, discussed in more detail, might clarify this.

Several bracket systems seem to have a nice correlation of a high value of the breakdown potential and a low nickel ion release (Smartclip™, SPEED™, TIME®). On the other hand further bracket systems exist where the behavior does not correlate to that extent (Damon® 2, Discovery®, In-Ovation®, Ultratrimm®). Looking onto the results in Table 3 it becomes obvious that a high breakdown potential might nevertheless result in high Ni ion release. Consequently we feel that an assessment of intraoral release of metal ions must be done on the basis of immersion tests. The electrochemical testing is an appropriate means to prepare a ranking of the overall corrosion susceptibility of the tested systems under clearly pre-defined *in-vitro* test condition.

Some special results have to be discussed for the Damon® 3 and the Opal™-M brackets. For the Damon® 3 system the low nickel release was to be expected, as the bracket body is made of composite and only the clip and slot were made of steel. So only a few ions were released from a small metallic surface. The highest breakdown potential of all brackets was measured for the Opal™-M brackets (1317 mV). This value correlates with a low nickel ion release of 0.30 μg/day. The nickel release is astonishing because the manufacturer prescribes this system as nickel free. It is possible that a false value was created by a measurement of doubly ionized tin (Sn) which can be mistaken for the nickel ion in mass spectrometer measurements.

The SEM analysis showed that the corrosion behavior of the surfaces differs between the bracket systems. In some cases the surface changes could be correlated with the measured nickel ion release. For example the Opal™-M showed good results in the electrochemical tests and also the surfaces had only slight corrosive lesions without pitting corrosion. Some other systems showed serious surface destruction (Damon® 2, Fig. 4A,B; Ultratrimm®, Fig. 8A,B), which could lead to a weakening of parts of the bracket structure.

In total, it has to be stated that the *in vitro* tests have several drawbacks in simulating the intra oral conditions. A bacterial bio film or a thermo cycling could not be tested in this study. Also a change of the pH level because of some foods or the mechanical loading was not simulated. Because of these facts it is reasonable to complete the results of this *in vitro* study with an *in vivo* investigation.

Conclusions and clinical relevance

The tested systems seem to be biocompatible and applicable for orthodontic therapy. The measured nickel release was far below the daily dietary intake level for all tested bracket systems. A static immersion test combined with the nickel ion release measurement seems to deliver information of high relevance for clinical application and biocompatibility while the electrochemical testing is a good means for classification and comparison of the systems with regard to corrosion resistance. In addition to the aspect of biocompatibility the corrosion might have clinically relevant effects on the surface microstructure. This change of the surface roughness might result in higher friction during the sliding of the bracket along the arch wire.

Clinical relevance

A static immersion test combined with the nickel ion release measurement seems to deliver information of high relevance for clinical application and biocompatibility while the electrochemical testing is a good means for classification and comparison of the systems with regard to corrosion resistance. In addition to the aspect of biocompatibility the corrosion might have clinically relevant effects on the surface microstructure. This change of the surface roughness might result in higher friction during the sliding of the bracket along the arch wire.

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