Original Article

Corrosion of orthodontic brackets: qualitative and quantitative surface analysis

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ABSTRACT

Objectives: To determine and compare surface characteristics and presence of corrosion in new and used brackets with optical light microscopy (OLM) and scanning electron microscopy (SEM), and with elemental chemical analysis with energy-dispersive X-ray spectroscopy (EDS).

Materials and Methods: OLM and SEM were used to analyze 24 new and 24 used conventional premolar brackets. EDS analysis was performed in six used brackets and four new brackets with corrosion-suspected spots.

Results: OLM and SEM images showed wear/abfraction signs, striations, pits/crevices, and adherent material. Used brackets showed more deterioration than new brackets. SEM images disclosed more morphological features than OLM images. EDS analysis revealed a significantly higher phosphorus ($P = .001$) and sodium ($P < .005$) weight fraction and significantly lower amounts of chromium ($P < .001$) in used brackets**.** The iron, chromium, and nickel weight fractions did not differ significantly between the clean and corrosion-suspected spots. Of the corrosion-suspected spots analyzed by combined SEM and EDS, 44.14% and 6.90% remained corrosion-suspected on used and new brackets, respectively.

Conclusions: Used brackets showed more signs of corrosion than new ones. Combined assessment of SEM and EDS indicates that the bracket surface is affected during orthodontic treatment as a result of corrosion. (*Angle Orthod.* 2022;92:661–668.)

KEY WORDS: Corrosion; Brackets

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INTRODUCTION

During orthodontic treatment, orthodontic brackets are exposed to hostile intraoral environmental challenges, such as humidity, pH/temperature fluctuations, mechanical forces, and plaque accumulation. These may cause brackets to deteriorate through the corrosion processes, but also due to frictional forces.[1,2]

Corrosion resistance depends on manufacturing procedures and a material's ability to form a protective surface (passivation) layer.[3] This is caused in stainless steel by oxide formation in the chromium and nickel. If this layer is disrupted, the corrosion process can start.[1,3,4]

Corrosion of orthodontic brackets has been found to affect orthodontic treatment by increasing surface roughness, which affects sliding mechanics by increasing friction.[2] It can also increase plaque accumulation and metal ion release, potentially with a toxic effect.[1,5,6]

Although previous studies used optical light microscopy (OLM) and scanning electron microscopy (SEM) to assess bracket corrosion solely on the basis of changes in color and surface morphology, these two

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Table 1. Composition of 3M Unitek Victory Series Low Profile Brackets as Provided by the Manufacturer (%)

Alloy	C	Mn	Si	Cr	Ni	Fe	*P*	S	Cu	Nb + Ta
17-4 PH	<0.07	<1	<1	17	4	~70	<.04	<0.03	4	0.3

criteria are not specific to corrosion, which must be confirmed by additional chemical analysis.[5–7] Because few studies that assessed corrosion combined energy-dispersive X-ray spectroscopy (EDS) with SEM to analyze chemical compositional changes in the bracket surface,[4,8] specific changes in the elemental composition of the corroded sites remain unclear.

The study had two main objectives: (1) to use OLM, SEM, and EDS analysis on new and used brackets to determine their surface characteristics and to compare any corrosion; and (2) to characterize and compare the elemental composition of corrosion-suspected spots with that of corrosion-free control spots.

It was hypothesized that: (1) the surface characteristics and morphology would not differ between used and new brackets; (2) the occurrence of corrosion would not differ between used and new brackets, and that (3) elemental compositions would not differ between corrosion-suspected and the corrosion-free control spots.

MATERIALS AND METHODS

This study was approved by the Ethics Committee at Academic Centre for Dentistry Amsterdam (ACTA) (#2020103).

In total, 48 conventional upper and lower second premolar brackets were analyzed (24 new and 24 used) (Unitek Victory Series Low Profile with APC adhesive, 3M Unitek, Monrovia, Calif.) with a 0.022 × 0.028-inch slot size. Used brackets were collected from 12 patients (six female, six male) who had been selected randomly from a pool of 460 patients treated between 2015 and 2018 with a full-fixed appliance at the Department of Orthodontics at the Academic Centre for Dentistry Amsterdam (ACTA). The used brackets were not subjected to any special cleaning procedure before intraoral placement. As practical conditions limited the sample size, the number of brackets studied was established on the basis of previous studies.[7,9–12] Mean patient age on the day of debonding was 18.4 years (range: 13.4–31.8). The mean treatment time was 23.5 months (range: 9.8–33.4). The brackets had been produced using the metal immersion molding technique (MIM).[12–14] Table 1 shows the composition of the brackets provided by the manufacturer.

Two brackets were selected from each patient, either from the first and the fourth quadrants, or from the second and the third quadrants. The distribution of the types of brackets was identical for new and used brackets.

Before analysis, all brackets were immersed in 50% acetic acid solution (CH_3COOH) for 72 hours, rinsed with distilled water, and then cleaned ultrasonically in the acetic acid solution and in distilled water, each time for 15 minutes. Finally, they were rinsed with distilled water and air-dried.

The overview images (Figure 1) and detailed images of the bracket bottom and cervical walls were taken with SEM at a 10 kV accelerating voltage (EVO LS15; Carl Zeiss, Oberkochen, Germany), and also with OLM (Stemi SV 6; Carl Zeiss). For the SEM images, the samples were fixed with conductive carbon adhesive tape on an aluminum stub. Finally, the OLM images were digitally magnified to match the magnification of the SEM images.

Using SEM and OLM images, 10 brackets were selected (six used, four new) with the most noticeable corrosion-suspected spots for EDS analysis. The following spots were suspected of corrosion: red-brown discoloration (on OLM); and striking irregularities and

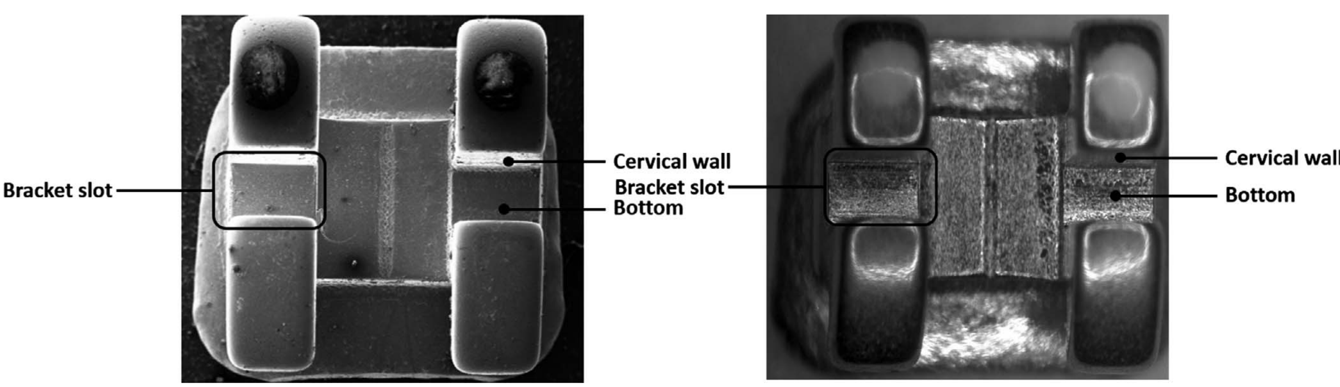

Figure 1. Same bracket, with SEM (mag. 60X) (left), and with OLM (mag. adapted to 60X) (right).

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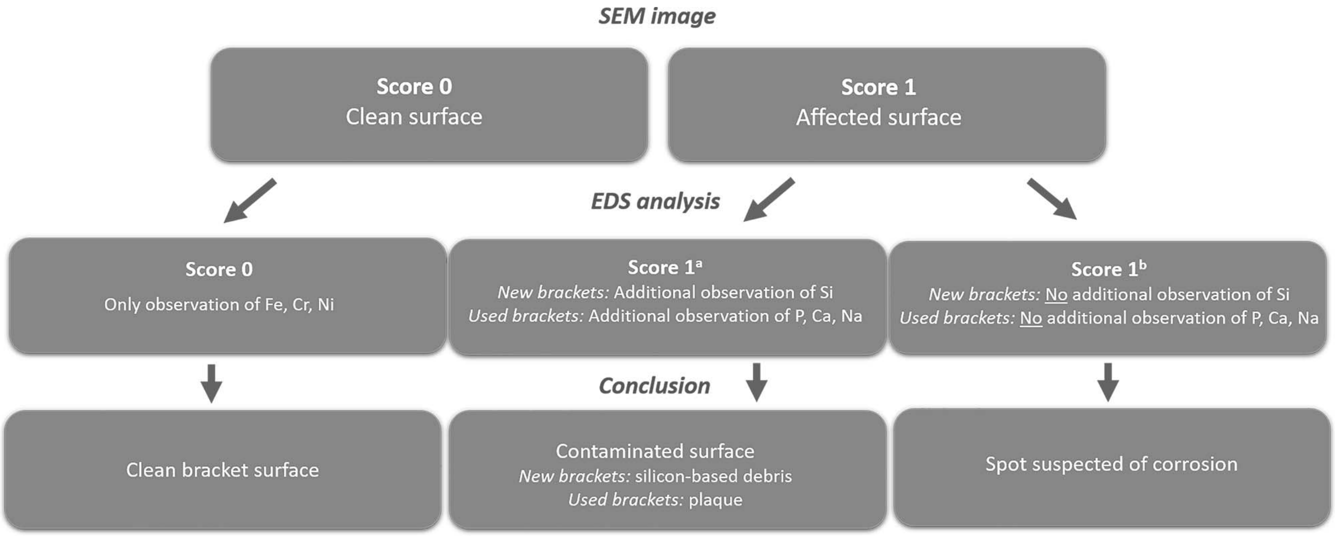

Figure 2. Qualitative classification (scoring system) of the spots.

pits/crevices (on SEM). For each bracket slot segment (left or right), corrosion-suspected spots and at least one control spot (ie, a spot without corrosion signs, debris, or plaque) were selected. If there were no corrosion-suspected spots on a bracket segment, a control spot from the same segment was selected. The EDS spectra of the selected regions of interest were obtained at a 20 kV accelerating voltage and 300 times magnification (Quantax XFlash 6130; Bruker, Billerica, Mass.).

Scoring of the Bracket Corrosion

On the basis of the SEM images and the EDS analysis, a scoring system was developed to determine whether the corrosion-suspected spots visible on the images could be regarded as (a) corrosion or (b) surface contamination (plaque or other debris); and whether control spots were indeed clean surfaces.

First, the spots on the SEM images that were selected for the EDS analysis were scored by three examiners (two orthodontists, and one dentist specialized in dental materials). Spots with visible corrosion/contamination were assigned a score of 1, and visibly clean spots were assigned a score of 0. In cases of doubt, agreement was reached between the examiners.

After the EDS analysis, the spots were further assigned to three categories. Spots on a visibly clean surface (score of 0) that contained only Fe, Cr, and Ni were assigned a score of 0 and were considered to be clean. Spots on a visibly affected surface (score of 1) containing Si, P, Ca, or Na were assigned a score of 1^a, and were considered to have been contaminated either with dental plaque during treatment (used brackets), or during the manufacturing process with Si-based debris (new brackets). Finally, if no Si, P, Ca, or Na were detected in these spots, a score of 1^b was given, ie, corrosion-suspected (Figure 2).

Statistical Analysis

Statistical analysis was performed with SPSS Statistics (Version 26.0) (IBM, Armonk, N.Y.). The elemental weight fractions in analyzed spots were compared between new and used brackets with an independent samples *t*-test. The paired samples *t*-test was performed to determine whether there was a significant difference between the mean weight fractions of the clean surfaces (score of 0) and the corrosion-suspected sites (score of 1^b). All tests were performed at a significance level of $P < .05$.

RESULTS

Microscopic Analysis

The morphological features noticeable on the bracket surfaces (Table 2) were wear/abfractions (small

Table 2. Overview of the Frequency of New and Used Brackets Showing Given Morphological Features Based on OLM and SEM Images

Images	Brackets	Wear and Abfraction, n (%)	Discoloration, n (%)	Striations, n (%)	Pits/Crevices, n (%)	Adherent Material, n (%)
OLM	Used	24 (100)	21 (87.5)	18 (75)	23 (95.8)	13 (54.2)
	New	1 (4.2)	11 (45.8)	18 (75)	16 (66.7)	13 (54.2)
SEM	Used	24 (100)	24 (100)	21 (87.5)	21 (87.5)	23 (95.8)
	New	1 (4.2)	20 (83.3)	23 (95.8)	16 (66.7)	24 (100)

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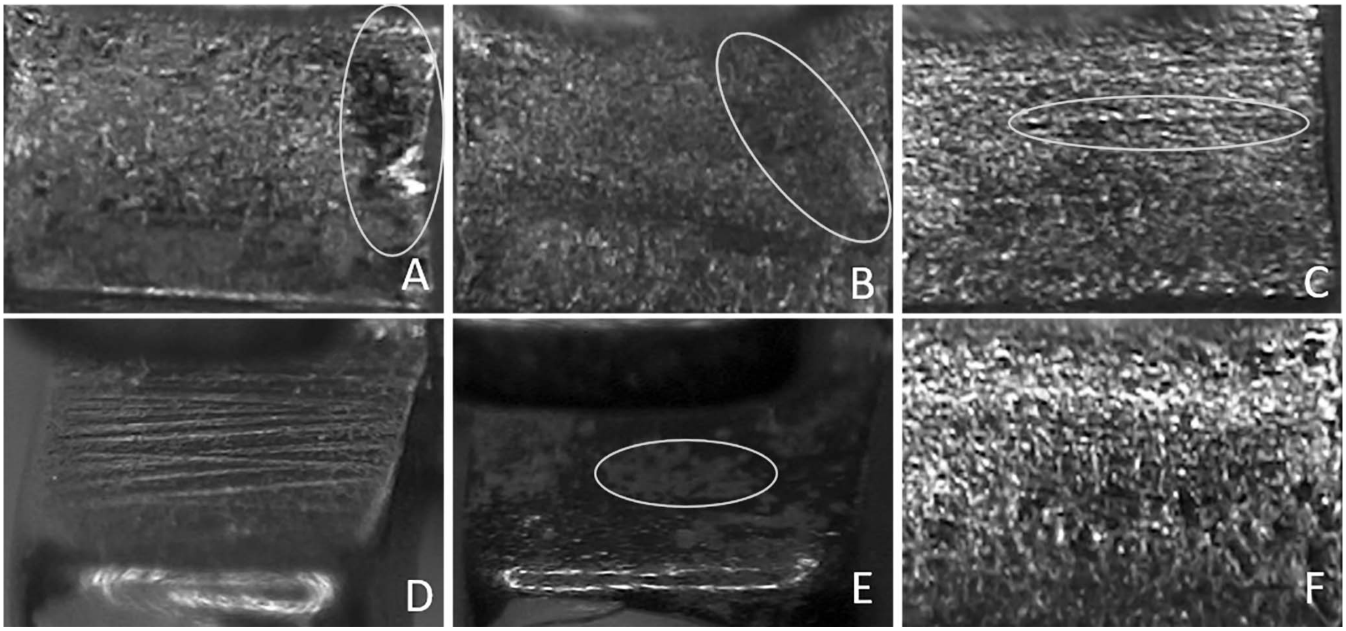

Figure 3. Morphological features (circled) visible on OLM images of used brackets (A–E) (mag. 60×) and on new brackets (F); (A) wear/abfraction, (B) discoloration, (C) pits/crevices, (D) striations, (E) adherent material, (F) bottom.

fragments of bracket crumbling off from the slot edges) (Figures 3A, 4A); discoloration (Figures 3B, 4B); pits/crevices (Figure 3C, 4C); striations (Figures 3D, 4D); and an adherent material (Figures 3E, 4E). SEM analysis showed more morphological features than OLM analysis, which showed more pits and crevices in used brackets (Table 2). Wear/abfractions were observed in all used brackets. Possible signs of corrosion were observed in only one new bracket. OLM images showed adherent material on used and new brackets as a blurry grayish-white color; SEM showed it as gleaming spots (Figures 5, 6).

EDS Analysis

Chromium, nickel, and iron were detected in all used and new brackets. No new brackets contained phosphorus. The chromium fraction was significantly

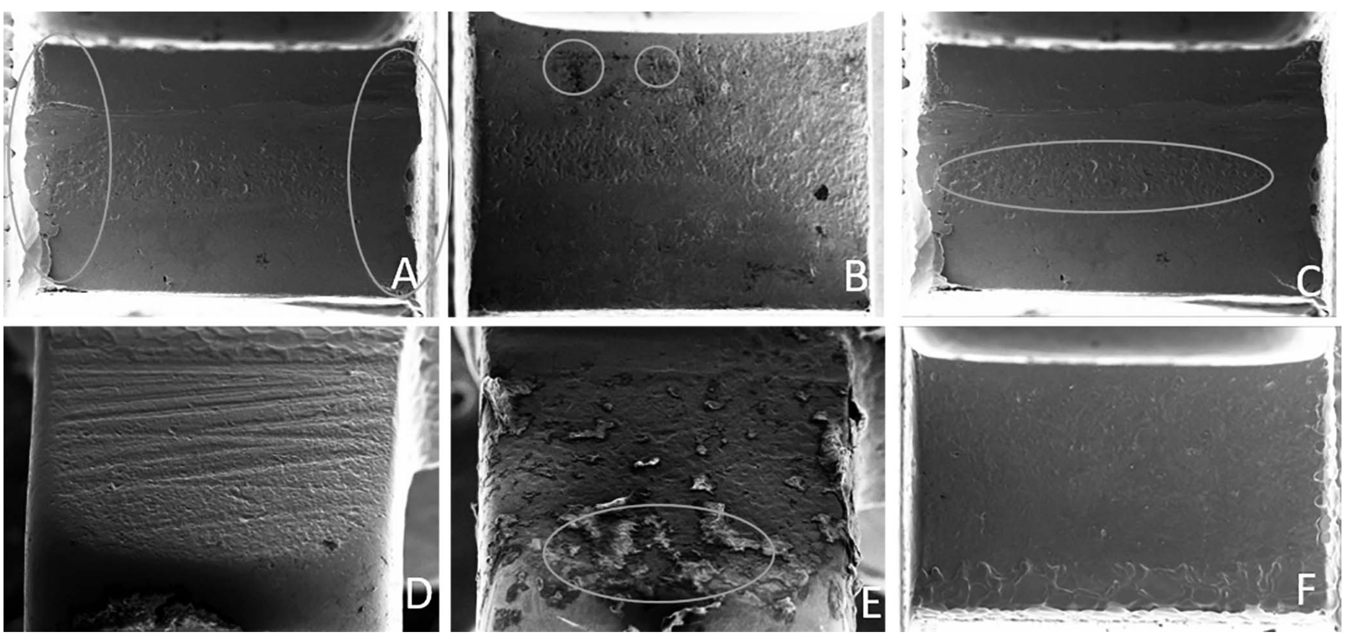

Figure 4. Morphological features (circled) visible on SEM images (300× magnified) of used brackets (A–E) and on new brackets (F); (A) wear/abfraction, (B) discoloration, (C) pits/crevices, (D) striations, (E) adherent material, (F) bottom.

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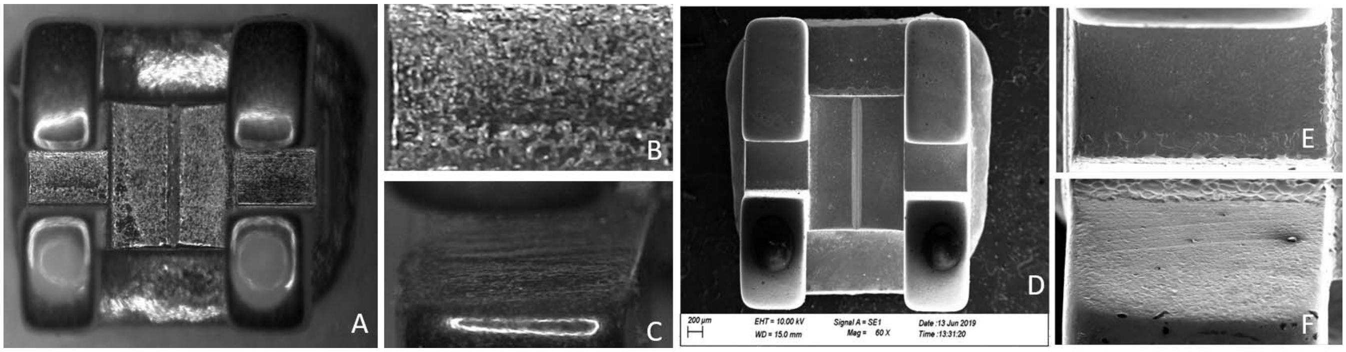

Figure 5. OLM (A–C) and SEM (D–F) images (image D 60×, images E&F 300× magnified, new bracket) (A, D) overview; (B, E) the bottoms of the bracket slots; (C, F) cervical walls.

higher in new brackets ($P < .001$), while the phosphorus ($P = .001$) and sodium ($P = .034$) fractions were significantly higher in the used brackets (Table 3). No significant differences were found between used and new brackets in the iron, nickel, and calcium fractions.

Additional elements generally absent in the standard bracket composition were detected in new and used brackets (Table 4). In used brackets, aluminum, silicon, zinc, tin, and titanium were detected in small weight fractions. The most apparent element in the new brackets was silicon, which was detected in 26 out of 28 contaminated spots (mean weight fraction: 59.14 ± 30.95%). Small amounts of nitrogen, fluoride, chlorine, potassium, and magnesium were also detected in all used brackets.

According to the qualitative assessment based on SEM images (step 1), 53 spots were free of corrosion or contamination, and were thus assigned a score of 0. A total of 116 spots were assigned a score of 1, ie, corroded or contaminated. After the EDS analysis, combined evaluation of SEM images and EDS analysis (step 2) showed that 51 spots remained that were corrosion or contamination free (score of 0), meaning that two originally clean spots appeared to have been contaminated after EDS assessment. Sixty-five spots were contaminated with plaque or silicon-based particles/debris (score 1[a]), and 53 were still corrosion-suspected (score of 1[b]), (Table 5; Figure 7).

Comparison of the mean composition of corrosion-suspected spots (N = 53, score of 1[b]) and clean spots (N = 51, score of 0) showed no significant differences in the mean weight fractions of iron, chromium, and nickel ($P > .05$). Neither were any significant differences found between the clean and corrosion-suspected spots on new and used brackets (Table 6).

DISCUSSION

OLM and SEM images of used and new brackets showed surface irregularities of various extent and degree. Used brackets had more surface irregularities that probably indicated surface deterioration related to orthodontic treatment and oral exposure.[3,7] However, the irregularities on new brackets indicated that their surface quality may have been affected by the manufacturing process itself.[7,15] In recent years, most brackets have been produced using metal injection molding (MIM), which is more cost-effective than preceding techniques, but results in surface irregularities.[12,16] These irregularities promote plaque retention

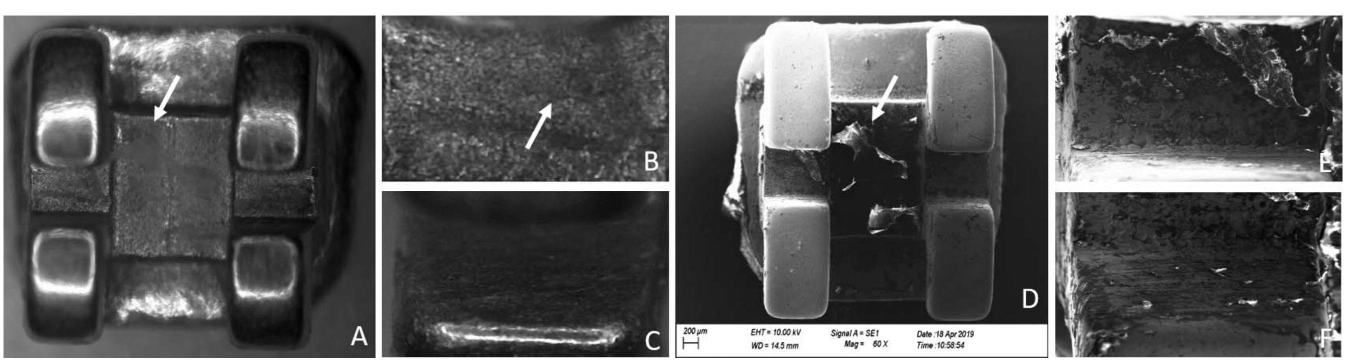

Figure 6. OLM (A–C) and SEM (D–F) images (image D 60×, images E&F 300× magnified, used bracket) (A, D) adhesive material visible in the middle bracket area (white arrow); (B, E) the bottoms of the bracket slots with visible red-brown discoloration (white arrow), wear, and deformation of the external edges; (C, F) cervical walls.

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Table 3. Mean Weight Fractions (%) of the Detected Elements in All Analyzed Spots of Used and New Brackets, With Significant Differences

	Used Brackets (N = 6, 111 spots)		New Brackets (N = 4, 58 spots)		
Element	Mean	SD	Mean	SD	*P* Value
Fe	71.48	21.89	77.02	14.98	.086
Cr*	11.30	6.94	13.79	3.63	.000
Ni	6.94	12.17	8.53	13.81	.443
P*	6.08	13.05	0.00	0.00	.001
Ca	1.15	1.15	0.05	0.34	.092
Na*	3.03	8.21	0.62	3.56	.034

* $P < .05$.

and create an environment favoring an anode-cathode reaction, possibly leading to corrosion.[3] The OLM images showed a red-brown discoloration, and the SEM images showed dark pits/crevices, which are features that correspond to different corrosion forms such as pitting, crevices, and stress corrosion in orthodontic alloys.[1,3]

While SEM images revealed more details than OLM images, neither technique was able to determine the chemical characteristics of the brackets or to differentiate between corrosion and adherent material (ie, plaque and debris). Because dark, irregular areas on the surface may stem from adherent material but also from corrosion, a complementary EDS analysis was also used, which showed a significantly lower Cr content, and lower Fe and Ni contents (Table 3) on used brackets. This indicated corrosion by the nickel

Table 4. Amount of Spots With Additional Elements Detected With EDS Analysis Generally Absent in the Standard Bracket Composition

Element	Used Brackets (N = 6, 111 spots)	New Brackets (N = 4, 58 spots)
Al	9	1
Si	9	26
Zn	8	0
Sn	8	0
Ti	10	0

and chromium released during treatment.[17–20] Though it was therefore expected that the chromium, nickel, and iron weight fractions would be lower at the corrosion-suspected sites than on the clean surfaces, they were not significantly lower (Table 6). Corrosion, as found in this study, might be a more generalized process that is characterized mainly by moderate loss of Cr, and is not related only to deteriorated surface sites.[21]

In used brackets, EDS analysis found P, Ca, and Na (Table 3); Al, Si, Zn, and Ti (Table 4); and, also, small amounts of N, F, Cl, K, and Mg, thereby showing that the cleaning procedures used had not completely removed the biofilm. Another study also found small amounts of plaque on used, thoroughly cleaned brackets.[7] On used brackets, Mikulewicz et al. detected Na, P, Cl, and K in areas less accessible to cleaning.[4,7] As a study by Lindel et al. found more biofilm accumulation on second premolar brackets than on canine and incisor brackets,[22] it was expected that more corrosion would be found on second premolar brackets in the current study.

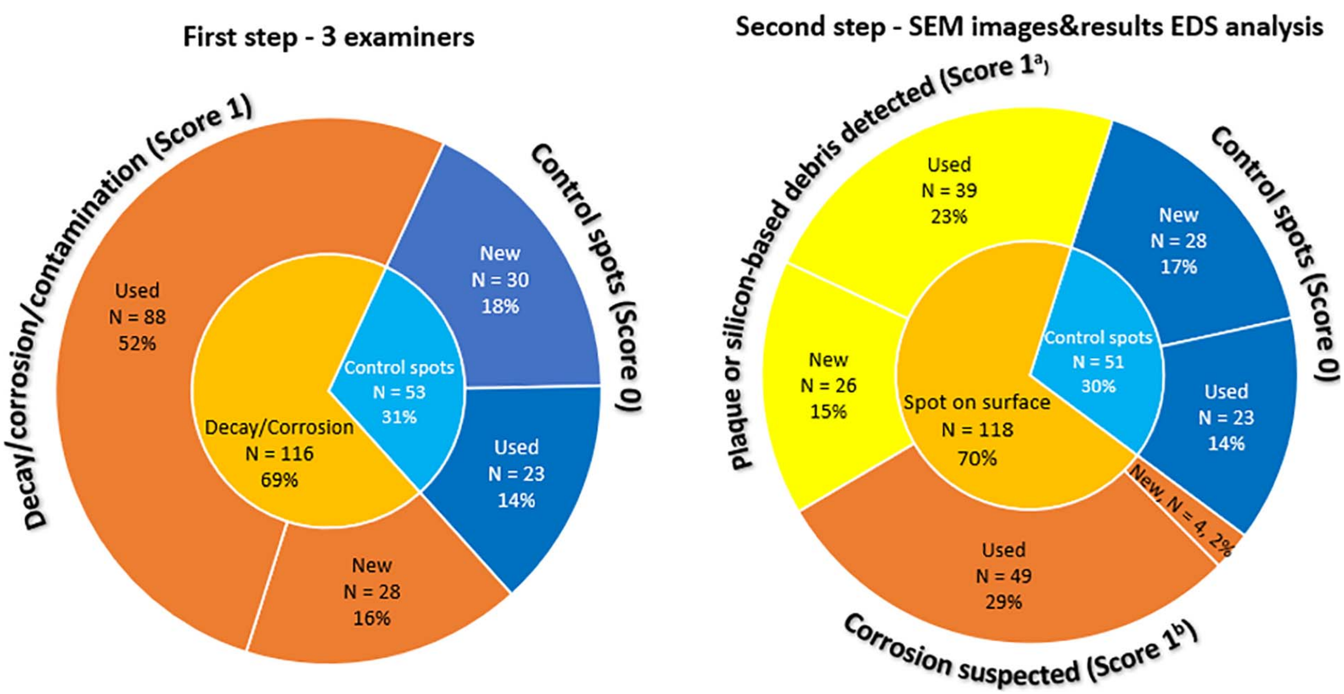

Figure 7. Qualitative surface assessment discriminating between control spots, contaminated spots, and corrosion-suspected spots.

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Table 5. Results of the Combined (SEM and EDS) Qualitative Assessment of the Selected Bracket Spots

Brackets	Analyzed Spots	Clean Surface (Score: 0), n (%)	Plaque (used) or Silicon-Based Debris (New) (Score: 1[a]), n (%)	Suspected of Corrosion (Score: 1[b]), n (%)
Used	111	23 (20.72)	39 (35.14)	49 (44.14)
New	58	28 (48.28)	26 (44.83)	4 (6.90)
Total	169	51 (30.18)	65 (38.46)	53 (31.36)

As Al, Zn, Sn, and Ti are not generally part of the bracket composition or of oral biofilm, exposure to these elements may occur during orthodontic treatment. Al and Zn are likely to originate from beta-titanium wires, and Ti from NiTi or beta-titanium wires.[3,23–25] Si, which is detected mainly in new brackets, may originate from bracket adhesive, the manufacturing process, and ligating elastics.

Although EDS improved differentiation between corrosion-suspected spots and spots with plaque or silicon-based debris, plaque or debris may impede reliable corrosion assessment. Similarly, evaluation of combined SEM and EDS analysis may underestimate the extent of any corrosion caused by undetected corrosion under plaque-covered spots (38% of the brackets).

Other studies may have used more aggressive cleaning procedures.[7,26] If so, it is unclear whether corrosion (or early signs of it) was also removed. For this reason, a second cleaning procedure to remove plaque remnants was not performed.

Plaque adhesion may favor the initiation of corrosion. But although biofilm formation reduces oxygen levels, thereby reducing the regenerative capacity of the passivation layer,[1,27] any causal relationship between plaque and corrosion could not be identified. Surface irregularities may, nonethless, promote plaque accumulation.

As the SEM and EDS analyses were so time-consuming, only one bracket type could be studied. As manufacturers using the MIM technique have several different production procedures,[12–14] studying more than one type would have complicated comparison of corrosion outcomes.

Several other studies[7,8] suggested that length of treatment would not affect the extent of corrosion. Though only a small number of brackets were tested, it was expected that their exposure to the oral environment would have been long enough to cause sufficient corrosion.

Study Limitations and Recommendations for Further Studies

Use of SEM to select affected spots and control spots for EDS analysis increased the risk of over-selection of corrosion-suspected spots more than random selection did.

Although the cleaning procedure used was more extensive than that in previous studies,[28,29] it was still not enough to remove all debris. This may have limited the detection of corroded spots. Further studies should develop more efficient cleaning procedures.

The cross-sectional study design precluded pretreatment bracket evaluation. To distinguish between the effect of the manufacturing process and orthodontic treatment on surface irregularities, pretreatment and post-treatment analyses of the same brackets are needed.

CONCLUSIONS

- Surface assessment with SEM and EDS analyses provides further evidence that orthodontic treatment causes corrosion and wear/abfraction of the bracket surface.
- Despite its limitations, the study provides more detailed evidence that new brackets have a rougher surface than manufacturers advertise.
- Plaque and biofilm adhering to the bracket surface thwarted corrosion assessment in many places.

Table 6. Comparison of the Mean Weight Fractions (%) of the Selected Elements Between the Corrosion Suspected Sites (N=53, Score: 1b) and Clean Sites (N-51, Score: 0) of 6 Used and 4 New Brackets, Based on the Combination of SEM Images and EDS Analyses

		Clean Sites (Score: 0)		Corrosion-Suspected Sites (Score: 1[b])		
	Element	Mean	SD	Mean	SD	*P* Value
Used brackets	Fe	83.02	6.65	81.64	6.44	.525
	Cr	11.28	0.99	11.79	0.85	.140
	Ni	5.68	6.40	6.58	6.62	.665
New brackets	Fe	82.30	9.24	87.64	2.33	.508
	Cr	12.46	0.26	12.36	2.33	.953
	Ni	5.24	9.07	0	0	.423
Total	Fe	82.78	7.01	83.64	6.03	.749
	Cr	11.67	0.99	11.98	1.37	.532
	Ni	5.53	6.80	4.38	6.18	.618

$P < .05$.

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- By distinguishing between plaque-contaminated and possibly corroded spots on used brackets, EDS eliminated the suspicion of corrosion on almost 50% of the spots shown by OLM and SEM.

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