British Journal of Medicine & Medical Research
2(4): 662-675, 2012

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Morphological and Structural Characterization of Two Types of As-Received and *In vivo* Orthodontic Stainless Steel Brackets

Sofie Hoste[1], Jean-Pierre Celis[2] and Guy Willems[1*]

[1]Department of Oral Health Sciences – Orthodontics, KU Leuven & Dentistry, University Hospitals Leuven, Belgium.
[2]Metallurgy and Materials Engineering, Faculty of Engineering, KU Leuven, Belgium.

Authors' contributions

This paper resulted from a joint collaboration at KU Leuven between dentists (authors SH and GW) and a material engineer (author JPC). Most of the experimental data were acquired by the author SH at the two participating KU Leuven departments. All the authors contributed to the writing of this paper, and they approved the final manuscript.

Research Article

Received 24th July 2012
Accepted 21st October 2012
Published 10th November 2012

ABSTRACT

Aims: To perform a) a topographical analysis of the bracket slot of 12 as-received stainless steel brackets and of *in vivo* used stainless steel brackets from 7 patients and b) a compositional characterization of the constituting materials of two types of brackets.
Study Design: In vitro laboratory study.
Methodology: 12 as-received brackets and *in vivo* used brackets of seven patients were analysed. Two commercially available brackets were used for this study. A SEM analysis evaluated the topographic features of each bracket slot while an EDAX analysis was performed to analyse the constituting parts of each bracket.
Results: As-received ORMCO brackets show more traces of plastic deformation than GAC brackets. On the contrary, pits, crevices and grain boundaries were observed on all as-received brackets. An increased plastic deformation and amount of scratches was noticed on ORMCO and GAC brackets after orthodontic treatment. Elemental analysis revealed that base and wing materials were not identical in GAC nor in Ormco brackets. In addition, both ORMCO and GAC brackets consist each of two parts assembled using a different brazing material.
Conclusion: Both as-received brackets show different damage patterns on their slot

**Corresponding author: Email: guy.willems@med.kuleuven.be;*

surface. Increase of damage patterns was visualized after intra-oral use. As a consequence of the use of different constituting materials for the manufacturing of the two types of brackets investigated, galvanic corrosion is likely to occur.

Keywords: Brackets; morphological evaluation; surface characteristics; as received; in vivo.

1. INTRODUCTION

Contemporary orthodontic treatment mostly involves the use of bonded brackets. Although these brackets can be manufactured from different materials, stainless steel is most commonly used, but ceramic, polymeric and titanium brackets are also available on the market (von Fraunhofer, 1997). Stainless steel is a family of steel based alloys containing chromium and nickel as main alloying elements. The main property of these alloys is a high corrosion resistance in aggressive water-based environments. Stainless steel can be divided into four main families according to their metallographic structure, namely austenitic, martensitic, ferritic and duplex (austenitic – ferritic) ones (Davis, 2003). Austenitic stainless steel has a face-centered cubic structure, is nonmagnetic in the annealed condition, and can be hardened by cold working. Austenitic stainless steel has good corrosion properties, contains a low carbon content (<0.1%), a chromium content between 16% and 26%, and a nickel content between 4 and 22%. Nickel stabilizes the austenitic structure. Martensitic stainless steels are essentially Fe-Cr-C alloys that posses a body-centered tetragonal crystal structure. The chromium and carbon content are balanced to ensure the martensitic structure. This family is ferromagnetic but its corrosion resistance is less than that of the austenitic family. The high hardness of martensitic stainless steels makes them suitable for the manufacturing of dental and surgical instruments. Ferritic stainless steel has a body centered crystal structure and contains between 13 and 18% chromium and less than 0.1% carbon. There is no nickel in ferritic stainless steel. Unlike martensitic stainless steel, ferritic stainless steel cannot be hardened by heat treatment. Ferritic stainless steel has few applications in medical devices. Duplex stainless steels contain approximately equal proportions of austenitic and ferritic phases in their microstructure. The main advantages of duplex stainless steel are the higher strength, an improved ductility, a better toughness and superior corrosion resistance compared to other types of stainless steel families (Platt et al., 1997; Davis, 2003). Orthodontic brackets are predominantly made out of austenitic stainless steel due to its good corrosion resistance, excellent formability, and low cost in comparison with the other types of stainless steels (Eliades et al., 2001).

Corrosion is an electrochemical process that results in a material loss due to a reaction of the material with an aqueous solution like saliva. The composition of the aqueous environment determines the extent of the corrosion process. The corrosion resistance of stainless steel relies on the presence of a self-healing surface layer (passive film) consisting mainly of a dense chromium oxide. Depending on intra-oral fluctuations in terms of acidity, composition of the saliva, and presence of micro-organisms as well as mechanical degradation, this protective film can break down so that corrosion is induced (Eliades and Athanasiou, 2002). Recently, it was shown that a stannous fluoride mouthwash may cause a considerable corrosion of stainless steel brackets through the destruction of the passive layer (Schiff et al., 2005). On the other hand, some salivary components such as amylase can act as a corrosion inhibitor (von Fraunhofer, 1997). Corrosion causes impairment of the mechanical properties, and increases the risk of failure. In the particular case of an orthodontic bracket and an inserted wire, the corrosion process can be enhanced by mechanical wear, which can cause corrosion-wear also known as tribocorrosion leading to a material degradation (Schiff et al., 2005). Another important aspect of corrosion of metallic alloys in oral fluids, is the micro-leakage of corrosion products like nickel ions in the case of austenitic stainless steel, in the human body. On patch testing, hypersensitivity due to nickel has been reported in up to 21.5% of the population (Shubert et al., 2005). Although there is evidence that nickel and its compounds can, at certain concentrations, cause harm when absorbed, it is not clear whether this readily occurs with orthodontic appliances (House et al., 2008).

The surface state of orthodontic brackets plays an important role for different reasons. First of all, a smooth arch wire bracket slot is essential to reduce friction (House et al., 2008). Secondly, a smooth surface reduces plaque deposition (Quirynen et al., 1994; Wheeler and Ackerman, 2009). Surface asperities are predilection spots for the onset of corrosion. The manufacturing process can play an important role in the surface topography of as-received stainless steel brackets. Therefore it is important to collect detailed information on the manufacturing process used by various commercial companies, but practice shows that such information remains limited notwithstanding its clinical importance.

The aim of this article is to report on the surface topography of the bracket slot of two types of commercial brackets in the as-received state and after *in-vivo* tests and to analyse the composition of the constituting parts of these two brackets.

2. MATERIALS AND METHODS

2.1 Description of the Material

The two types of stainless steel brackets used in this study are the standard edgewise bracket of GAC (GAC Dentsply, NY, USA) and the mini twin bracket of ORMCO (ORMCO Corporation, Glendora, CA, USA). Twelve as-received brackets were examined as well as brackets of seven patients collected after careful de-bonding. For each patient, 4 brackets were analysed, namely at positions 11, 14, 31 and 33. All brackets were thoroughly cleaned. For some patients, ORMCO and GAC brackets were simultaneously used, while for other patients, only GAC or ORMCO brackets were placed. This bracket distribution (Table 1) did not happen at random, but depended on patient's characteristics and the personal preference of the practitioner. Because of the smaller mesiodistal dimensions of lower front ORMCO brackets, these lower incisor brackets were used in patients with small teeth. Upper incisor ORMCO brackets have an angulation of 5 degrees while GAC brackets do not have such a pre-adjusted angulation. Initial phases of treatment were accomplished using standard Nickel-Titianium wires whereas afterwards only custom made Stainless Steel wires were used.

2.2 Cleaning Procedure and Surface Analysis

After careful de-bonding, brackets were stored in alcohol for 24h. These in vivo tested as well as as-received brackets were ultrasonically (US) cleaned. A first US cleaning was performed in an alkaline solution (VR 6334-16, Henkel, Brussels, Belgium) at 60ºC for 15' to remove organic substances (e.g. plaque). After rinsing with distilled water at room temperature, the specimens were US cleaned with a 4% sulphuric acid solution to remove inorganic debris (e.g. calculus). Then the brackets were rinsed again with distilled water, and subsequently with ethanol (naturalized ethanol + 5% per cent diethyl ether) and dried with warm air. Daems et al. previously used this cleaning procedure (Daems et al., 2009).

The specimens were individually fixed in round sample holders, and sputter coated with a Au-layer of 10 nm (Balzers Union Sputtering Device, Balzers, Principality of Lichtenstein), which improves surface conductivity required for SEM-investigation. The samples were protected from dust with a plastic lit on each individual holder. Each specimen was observed by scanning electron microscopy (SEM, FEG-Philips XL-30, Philips Company, Eindhoven, The Netherlands) equipped for energy dispersive X-ray analysis (EDAX). Six sites were analysed namely all slot walls (tooth, gingival, and occlusal sides) on the mesial and distal wing. To eliminate the canting of the bracket for the observation of the gingival and occlusal side, some brackets were sliced in two pieces with a rotating diamond disc. Afterwards, the bracket was cleaned again ultrasonically with distilled water.

Table 1. Bracket distribution in each patient

	Patient's initials	**GAC**	**ORMCO**
	GB	14, 33	11, 31
	GP	11, 14, 33	31
	JT	11, 14, 33	31
	PL	11, 14, 31, 33	
	SF		11, 14, 31, 33
	SV	14, 31, 33	11
	VD		11, 14, 31, 33
Total	7	15	13

2.3 EDAX Analyses

To examine the elemental composition of the different parts of GAC and ORMCO brackets, microanalyses were performed using energy dispersive X-ray microanalyses (EDAX, threshold value of 0.8 wt %). Therefore, two as-received brackets of each bracket type, were sliced along the bracket slot, and subsequently embedded in an epoxy resin. Elemental analysis was performed on the bracket wings, the bracket bases and the intermediate brazing materials. Elemental analysis was also performed at selected areas on as-received and in vivo used brackets to determine the composition of certain surface defects visible on the bracket slots.

3. RESULTS AND DISCUSSION

3.1 Surface Features of As-Received Brackets

Some topographic features are always present on GAC as well as on ORMCO brackets whereas some other features are appearing now and then. A broad variety in the quality of the surface finish is also noticed on both types of brackets. Some brackets exhibit almost no surface defects, while others have defects to a great extent. All those features are summarized in Table 2.

Table 2. Surface characteristics of As-received brackets

GAC	pits	crevices	grain boundaries	plastic deformation	scratches	grooves on tooth surface	grooves on occl/ging surface	nucleation/ growth	honeycomb structure	wave like pattern
UI1	+	+	+	-	-	-	+	-	-	-
UI2	+	+	+	-	-	-	+	-	+	-
UI3	+	+	+	+	-	-	+	-	-	-
PM1	+	+	+	-	-	-	+	-	-	-
PM2	+	+	+	-	-	-	+	-	-	-
PM3	+	+	+	+	-	-	+	-	-	-

ORM CO	pits	crevices	grain boundaries	plastic deformation	scratches	grooves on tooth surface	grooves on occl/ging surface	nucleation/ growth	Honeycomb structure	Wave like pattern
LI1	+	-	-	+	-	-	-	-	+	-
UI1	+	+	+	+	+	+	+	-	-	-
UI2	+	+	+	+	+	+	+	-	-	-
PM1	+	+	+	+	+	+	+	-	-	+
PM2	+	+	+	+	-	-	-	-	+	-
PM3	+	+	+	+	-	-	+	-	-	-

Occl: occlusal, ging: gingival, UI: upper incisor, PM: premolar, LI: lower incisor

On As-received GAC brackets, pits, crevices, grain boundaries, and parallel grooves are always noticed on the occlusal and gingival slot surfaces (Fig. 1a). Plastic deformation at the slot border was obvious on two as-received GAC brackets. A honeycombed structure due to excessive pickling was noticed on one bracket. Not a single as-received GAC bracket showed grooves or scratches on the slot tooth surfaces.

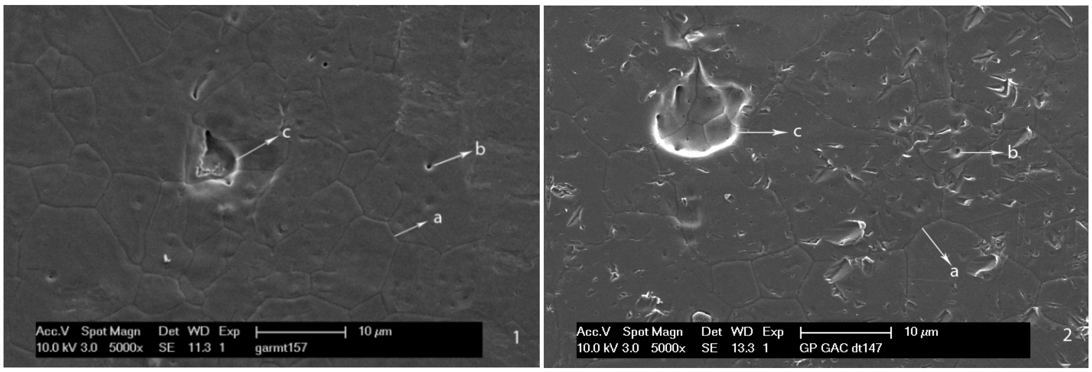

Fig. 1. As-received (1) and in vivo (2) GAC bracket. Visualisation of grain boundaries (a) pits (b) and plastic deformation (c). Plastic deformation tends to increase during intra-oral use

Pits and plastic deformation were noticed on all as-received ORMCO brackets (Fig. 2a). All except one as-received bracket showed crevices and grain boundaries. Scratches, grooves on the slot tooth surface, and grooves on the occlusal and gingival surfaces were obvious on three as-received brackets. Two as-received ORMCO brackets showed a honeycomb structure probably the consequence of an excessive pickling (Fig. 3). On two ORMCO brackets a wavelike pattern is visible on some grains. This wavelike pattern appears in different grains in different directions (Fig. 4).

3.2 Surface Features of Retrieved Brackets

Surface features noticed on in vivo used brackets are summarized in Table 3. After in vivo use, a plastic deformation is obvious on all except one retrieved GAC bracket. Compared to as-received brackets, a slight increase of the number of grooves on the tooth surface is noticed (Fig. 1b). The same observation was done on retrieved ORMCO brackets (Fig. 2b). Debris is present on the slot surface of some samples. On several in vivo used ORMCO and GAC brackets, nucleation of organic material at the grain boundaries is noticed (Fig. 5). On a number of in vivo used ORMCO brackets, the wavelike pattern was also observed (Fig. 4).

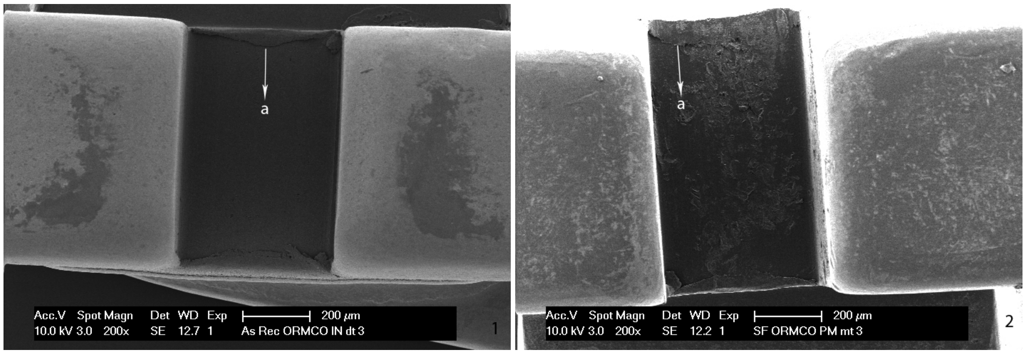

Fig. 2. As-received (1) and in vivo (2) ORMCO bracket. Plastic deformation (a) is already present on as-received sample at the slot border and tend to increase in in vivo sample on the entire surface

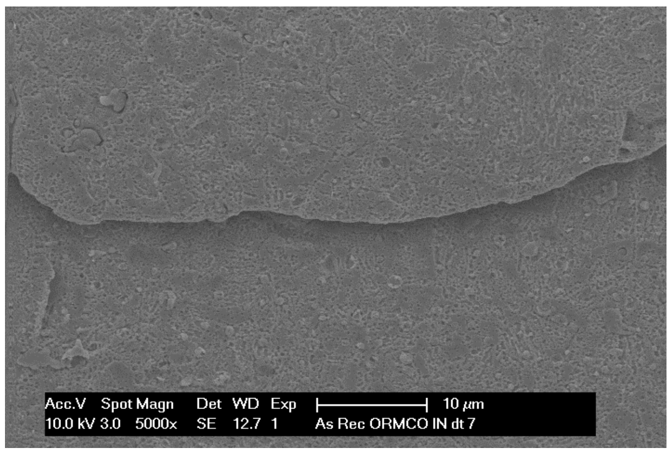

Fig. 3. As-received ORMCO bracket with bad pickling procedure and generalized corrosion

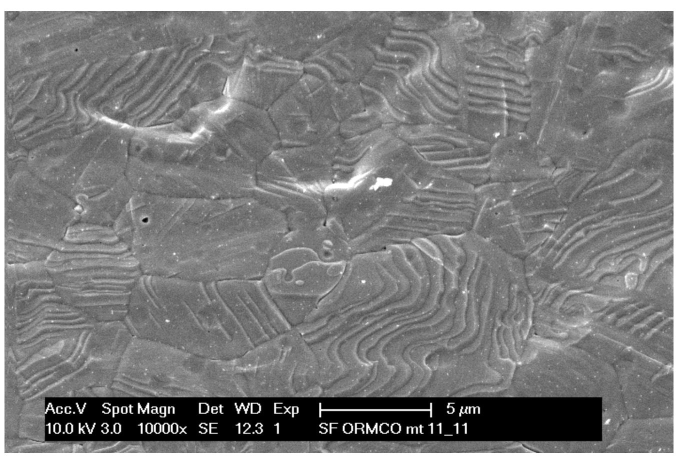

Fig. 4. *In vivo* ORMCO bracket with wavelike pattern on the tooth side of the slot.

Table 3. Surface characteristics of in vivo used brackets

GAC (patient's initials)	Pits	Crevices	Grain boundaries	Plastic deformation	Scratches	Grooves on tooth surface	Grooves on occl/ging surface	Nucleation/ growth	Honeycomb structure	Debris	Wavelike pattern
GP 11	+	+	+	-	-	-	+	+	-	-	no
GP 14	+	+	+	+	+	+	+	-	-	-	no
GP 31	+	-	+	+	+	-	+	+	-	-	no
JT 11	+	+	+	+	+	-	+	+	-	+	no
JT 14	+	+	+	+	-	+	+	+	-	-	no
JT 33	+	+	+	+	-	-	+	+	-	+	no
PL 11	+	+	+	+	+	-	+	-	-	-	no
PL 14	+	+	+	+	+	+	+	-	-	-	no
PL 31	+	+	+	+	+	-	+	-	-	-	no
PL 33	+	+	+	+	+	-	+	+	-	-	no
SV 14	+	+	+	+	+	-	+	+	-	-	no
SV 31	+	+	+	+	+	+	+	+	-	-	no
SV 33	+	+	+	+	+	-	+	+	-	-	no
GB 33	+	+	+	+	-	-	+	-	-	-	no
GB 14	+	+	+	+	+	-	+	-	-	-	no
ORMCO	**Pits**	**Crevices**	**Grain boundaries**	**Plastic deformation**	**Scratches**	**Grooves on tooth surface**	**Grooves on occl/ging surface**	**Nucleation/ growth**	**Honeycomb structure overpickling)**	**Debris**	**Wavelike pattern**
GB 11	+	+	+	+	+	+	+	-	-	-	yes
GB 31	+	+	+	+	+	-	+	-	-	+	no
GP 31	+	+	+	+	+	+	+	+	-	-	no
JT 31	+	+	+	+	+	+	+	+	-	+	no
SF 11	+	+	+	+	+	+	+	+	+	+	yes
SF 14	+	+	+	+	+	-	+	+	-	-	no
SF 31	+	+	+	+	+	+	+	+	+	-	no
SF 33	+	+	+	+	+	+	+	-	-	+	yes
VD 11	+	+	+	+	+	-	+	-	-	-	no
VD 14	+	+	+	+	+	+	+	-	-	-	no
VD 31	+	+	+	+	+	+	+	-	-	+	yes
VD 33	+	+	+	+	+	-	-	+	-	+	yes
SV 11	+	+	+	+	+	+	+	-	-	-	yes

Occl: occlusal, ging: gingival, UI: upper incisor, PM: premolar, LI: lower incisor

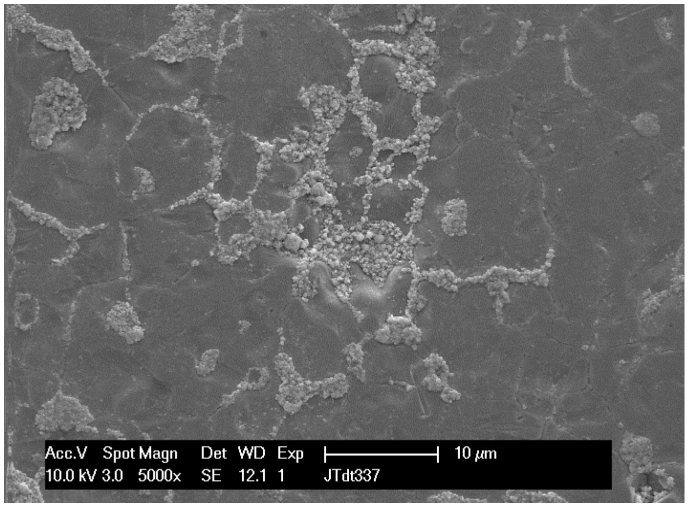

Fig. 5. *In vivo* GAC bracket with nucleations on the tooth side of the slot border preferentially at the grain boundaries

3.3 EDAX Analysis

Elemental analyses revealed that the bracket base and the wing part of ORMCO and GAC brackets are made of different alloys. Result of elemental analysis is listed in Table 4 and information obtained from the companies in Table 5. The GAC wing can be defined as a 303 SS material, and the GAC base as a 316 SS material. On the contrary, the ORMCO bracket wing consists of martensitic 17-4 precipitation-hardened SS material while the base is also austenitic 316 SS.

Table 4. Elemental composition of ORMCO and GAC bracket from EDX analysis (wt%)

Element	Si	Mo	Cr	Mn	Fe	Ni	Cu	Ag	Au
GAC wing	0.51 ± 0	0.99 ± 0.06	18.44 ± 0.13	1.78 ± 0.02	67.57 ± 0.05	9.82 ± 0.08	0.9 ± 0.03		
GAC base	0.67 ± 0.03	1.75 ± 0.12	16.9 ± 0.02	1.4 ± 0.05	66.1 ± 0.26	12.55 ± 0.07	0.7 ± 0.01		
GAC brazing material							25.47 ± 0.01	37.48 ± 1.44	37.0 ± 1.45
ORMCO wing	0.43 ± 0.07		15.23 ± 0.18	0.79 ± 0.09	75.85 ± 0.1	4.4 ± 0.2	3.32 ± 0.05		
ORMCO base	0.65 ± 0.03	1.75 ± 0.13	17.26 ± 0.08	1.25 ± 0.13	66.75 ± 0.03	12.36 ± 0.01			
ORMCO brazing material			2.41 ± 0.09		8.77 ± 0.46	12.57 ± 0.5			76.25 ± 1.04

Table 5. Elemental composition of the GAC bracket (wt%), information supplied by the company

GAC	C	S	Mn	P	S	Ni	Cr	Mo	Fe	Au	Cu	Ag
Wing	<0.15	<1	<2	<0.2	<0.15	8-10	17-19		balance			
Base	<0.08	<1	<2	<0.045	<0.03	10-14	16-18	2-3	balance			
Brazing material										33	28	39

The elemental composition of the brazing material used to connect base and wing parts was in the case of GAC 38.92 wt% Ag, 35.6 wt% Au, and 25.48 wt% Cu. In the case of ORMCO the composition was 77.29 wt% Au, 12.08 wt% Ni, 8.31 wt% Fe, and 2.31 wt% Cr. On GAC brackets, a clear boundary between brazing material, and the surrounding base and wing material is noticed. Sometimes, the wing material touches directly the base alloy with no interconnecting brazing material. This is not the case on ORMCO brackets where a diffusion of brazing material into base and wing materials, is noticed (Fig. 6). Backscattered electron images of the tooth side of the bracket slot on as-received GAC brackets revealed that a diffusion of brazing material took place, contaminating the surface of this tooth side with brazing material mainly at grain boundaries.

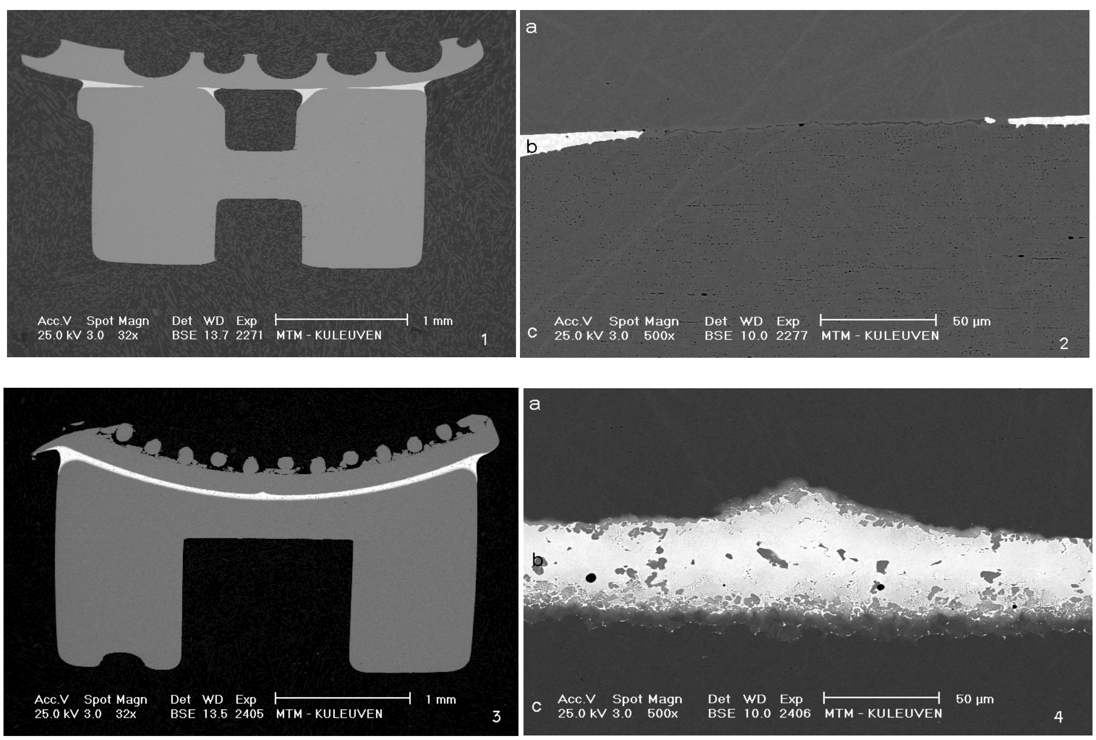

Fig. 6. Backscattered images of as-received GAC (1) and ORMCO (3) bracket, sliced along the bracket slot to visualise the different composing alloys of each bracket. Larger magnification of the brazing material. GAC bracket (3) shows a clear distinction between the base (a), wing (c) and brazing (b) material and sometimes a close contact between the base and wing alloy. In the ORMCO bracket (4), there is a diffusion of brazing (b) material in the surrounding base (a) and wing (c) alloy

Elemental analyses of certain surface defects noticed on in vivo used GAC and ORMCO brackets revealed the presence of remnants of organic constituents. Other defects contain elements composing the bracket wing.

No information was provided by ORMCO, even after numerous attempts. From GAC, the authors received a product material sheet. Table 5 shows the elemental composition of GAC brackets as received from the company.

3.4 Discussion

With this research, the authors succeeded in establishing a catalogue of possible surface defects observed on two commercial brackets.

As-received and *in-vivo* used brackets show surface defects to different extents. A smooth surface finish as claimed by some manufacturers appears from this investigation more as an exception than as a rule. Pits, crevices, grain boundaries, and plastic deformation were observed on all as-received ORMCO brackets investigated. As-received GAC brackets show less plastic deformation although pits, crevices, and grain boundaries appeared on all samples investigated. These defects must be considered as a consequence of the manufacturing process.

Three main shaping processes used to manufacture brackets are casting, machining or milling, or powder sintering.1 However, information provided by the manufacturers or available in open literature on the shaping process is very limited. In view of the damage patterns noticed on the occlusal and gingival sides of as-received brackets, a milling technique can be assumed.

During the high temperature shaping of stainless steels, the formation of an oxide scale is commonly accepted resulting in the formation of an underlying chromium-depleted layer. This oxide layer has due to its porous structure inferior protective properties compared to the native dense passive surface layer formed in contact with ambient air at room temperature. In order to restore the corrosion resistance of stainless steels shaped at high temperature, the oxide layer and the chromium-depleted layer need to be removed by chemical or electrochemical pickling. Pickling can consist of an anodic polarization in a neutral electrolyte, followed or not by a treatment at open circuit potential in an oxidizing acidic electrolyte intended to remove oxide and chromium-depleted surface layers. During this pickling, it is important to remove a minimum amount of bulk material (Li and Celis, 2003). If the pickling is not done properly, a risk of increased corrosion arises. Pickling in a solution containing a too low HCl-concentration can induce a localized corrosion resulting in pits, etched grain boundaries, and honeycombed recesses (Li et al., 2008). Such defects were indeed sometimes observed in this study at plastically deformed slot borders of as-received ORMCO brackets.

The assembling of the bracket onto the base by brazing is also a matter of major concern. Backscattered electron images of the bracket slot on as-received GAC bracket revealed clearly the contamination of the surface of the slot with brazing material. This brazing material is appearing mainly at grain boundaries. As a consequence, a galvanic cell can be established between the noble brazing material and the stainless parts once immersed in a water-based solution. This might cause a localized corrosion known as galvanic corrosion. Different brazing materials were used for assembling ORMCO and GAC brackets. GAC uses a Au-Ag-Cu brazing material, while ORMCO used a brazing material consisting mainly of Au.

The results obtained in this study for GAC brackets, are in agreement with a study done by Zinelis et al. (2004). They investigated the quantitative composition of four different brazing alloys by energy dispersive X-ray microanalysis. In the case of ORMCO brackets, they found less Au, namely 67, 79 wt%, than in this study (77, 29 wt %). Due to the fact that the brazing material in ORMCO brackets consists mainly of Au, there is no doubt that galvanic couples are established with the base and wing materials inducing a typical corrosion process. Which material would behave preferentially as anode is a matter of further research. ORMCO states in his commercial flyer that this Au-based brazing material is the reason for an optimal corrosion resistance and resistance to mechanical deformation. Indeed, Au is a noble material thus not sensitive to corrosion, but when connected to a less noble material such as the 17-4PH SS or 316 SS, galvanic coupling forces the less noble materials to dissolve.

On in vivo used GAC brackets, a plastic deformation and parallel grooves on the tooth surface were noticed. This might be attributed to a low dimensional tolerance between wire and slot. A study done by Flores et al compared the plastic deformation in 5 different bracket materials, and found that 303 SS and 17-4PH stainless steel had the highest yield strengths compared to 310, 303Se and 316L stainless steel (Flores et al., 1994). These differences in yield strength can be related to differences in microhardness reported by Eliades et al. (2003). He reported a lower hardness for the 316 base material of the Ormco bracket compared to a stiffer wing part being as in the 17-4 PH SS. These differences in hardness have clinical goals. Namely, a low hardness to facilitate debonding while a stiffer wing can withstand higher loads. Notwithstanding a high yield strength, the wings of ORMCO and GAC brackets are obviously subjected to plastic deformation.

The wavelike pattern observed on some as-received and in vivo used ORMCO brackets could result from a corrosive attack (Fig 4). A hypothesis could be that due to the fact that martensite is not a balanced structure in which energy is stored, it may suffer from an increased corrosion susceptibility linked to the release of that stored energy.

Since in this research the as-received and in-vivo used brackets that were investigated were not the same samples, there is no information available on the starting and ending conditions of a given sample. A further systematic investigation of that aspect is thus needed. Nevertheless, some interesting conclusions result from this investigation as summarized hereafter.

4. CONCLUSION

1. As-received brackets show a variety of surface defects that can be places where the risk of a corrosive attack is enlarged. Some brackets show relatively more traces of plastic deformation than do other from different manufacturer.
2. More traces of plastic deformation are noticed on in vivo used brackets compared to as-received brackets. Possible origins are either the handling by the dentist during bonding brackets and installing wires or their retrieval, or a mechanical interaction between wires and brackets during in vivo use e.g. during to tooth movement.
3. The different materials used for the assembly of both types of brackets generate a potential risk of an intrabracket galvanic corrosion causing the release of metallic ions into the saliva of the patients. The extent of this corrosion is still the subject of further research.

4. The mechanical interactions at contacting points between wires and brackets can cause a wear of the constituting materials resulting in solid debris that can be released in the saliva of patient. The extent of this wear loss needs to be clarified by further investigations.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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