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Sepp Hersche, Iosif Sifakakis, Spiros Zinelis and Theodore Eliades*

Elemental, microstructural, and mechanical characterization of high gold orthodontic brackets after intraoral aging

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Abstract: The purpose of the present study was to investigate the elemental composition, the microstructure, and the selected mechanical properties of high gold orthodontic brackets after intraoral aging. Thirty Incognito™ (3M Unitek, Bad Essen, Germany) lingual brackets were studied, 15 brackets as received (control group) and 15 brackets retrieved from different patients after orthodontic treatment. The surface of the wing area was examined by scanning electron microscopy (SEM). Backscattered electron imaging (BEI) was performed, and the elemental composition was determined by X-ray EDS analysis (EDX). After appropriate metallographic preparation, the mechanical properties tested were Martens hardness (HM), indentation modulus (E_{IT}), elastic index (η_{IT}), and Vickers hardness (HV). These properties were determined employing instrumented indentation testing (IIT) with a Vickers indenter. The results were statistically analyzed by unpaired t-test ($\alpha=0.05$). There were no statistically significant differences evidenced in surface morphology and elemental content between the control and the experimental group. These two groups of brackets showed no statistically significant difference in surface morphology. Moreover, the mean values of HM, E_{IT} , η_{IT} , and HV did not reach statistical significance between the groups ($p>0.05$). Under the limitations of this study, it may be

concluded that the surface elemental content and microstructure as well as the evaluated mechanical properties of the Incognito™ lingual brackets remain unaffected by intraoral aging.

Keywords: hardness; instrumented indentation testing; lingual; orthodontic brackets; scanning electron microscopy; surface analysis.

Introduction

Nowadays, the number of adults seeking orthodontic treatment has increased considerably. The demand for aesthetically pleasing orthodontic appliances has grown, and this topic has become the subject of extensive research and appliance development. Removable thermoplastic appliances are nearly “invisible” and widely used; however, their major biomechanical limitations must be borne in mind when choosing this type of appliance. Lingual orthodontics, which was invented in the 1970s by the Japanese orthodontist Fujita [7], is the sole alternative for treating patients with an almost invisible appliance. Incognito™ (Figure 1), launched in 2004 [3], is a popular fully customized lingual appliance system. These brackets are manufactured from a nickel-free high gold alloy, suitable for use in treating patients with nickel allergies [5, 15].

Previous data suggest that both Cu and Zn are massively dissolved from silver-based soldering alloys used in orthodontics [6] and pediatric dentistry [13], raising serious biological concerns about oral lesions and allergic reactions [2, 6, 11]. In addition, Au-Cu-Ag alloys are prone to a very specific degradation process commonly known as “dealloying” or “selective leaching”, a process where one or more less noble metals are selectively dissolved during functioning. Cu and Ag are both dissolved from Au-Cu-Ag alloys in human saliva [14] and even from amorphous Au-Cu alloys [10]. Two theories have been proposed for this phenomenon. According to the first, the two metals in an alloy are dissolved in the surrounding media, but somehow one redeposits on the alloy surface. According to the other theory, one element is selectively dissolved from

***Corresponding author: Theodore Eliades**, Clinic of Orthodontics and Paediatric Dentistry, Center of Dental Medicine, University of Zurich, Plattenstrasse 11, CH-8032 Zurich, Phone: +41 44 634 32 10/11, Fax: +41 44 634 43 35, E-mail: theodore.eliades@zzm.uzh.ch

Sepp Hersche: Clinic of Orthodontics and Paediatric Dentistry, Center of Dental Medicine, University of Zurich, Zurich, 8032, Switzerland

Iosif Sifakakis: Department of Orthodontics, School of Dentistry, University of Athens, Athens 11527, Greece

Spiros Zinelis: Clinic of Orthodontics and Paediatric Dentistry, Center of Dental Medicine, University of Zurich, Zurich, 8032, Switzerland; and Department of Biomaterials, School of Dentistry, National and Kapodistrian, University of Athens, Athens, 11527, Greece

Figure 1: Incognito™ lingual brackets bonded on the lower arch.

the surface leaving a porous residue enriched in noble elements [14]. The mechanical properties of the appliance are affected by the dealloying process too, as the alloy volume becomes porous. The clinical implications are twofold – bracket surfaces should be hard enough to resist deformation and wear during their function, and bracket alloys should be stiff enough in order to avoid plastic deformation during ligation.

The golden lingual brackets have been used in orthodontic clinical practice since 2002, but there is no information available for the possible alterations of their elemental or mechanical properties after intraoral use. The aim of this study was to investigate the effect of intraoral aging on high gold orthodontic brackets of the Incognito™ system, referring to elemental, microstructural, and mechanical properties. The null hypothesis is that significant alterations in the elemental and mechanical properties of these brackets are anticipated after intraoral aging.

Materials and methods

Sample collection

Fifteen brackets (Incognito™, Bad Essen, Germany) were retrieved from different patients after the end of orthodontic treatment (INV). The mean time of intraoral aging was 23 ± 2 months. A set of 15 new brackets was used as reference (REF). This study was approved by the Research Ethics Committee of the University of Athens, Greece.

Scanning electron microscopy/EDS analysis

All brackets were investigated by scanning electron microscopy (SEM) (Quanta 200, FEI, Hillsboro OR) and their surfaces were imaged employing backscattered electron emission at 12,000X nominal magnification under high vacuum conditions (chamber pressure 10^{-6} Torr) with an accelerating voltage 20 kV and beam current 105 μ A. The elemental composition of brackets was identified and determined by X-ray energy-dispersive spectrometry (EDX). One X-ray EDS spectrum was collected using an X Flash 6|10 Silicon Drift Detector (Bruker, Berlin, Germany) from the wing of each bracket (Figure 2) employing the aforementioned high tension conditions 300X nominal magnification with a $430 \times 430 \mu\text{m}$ collecting window. The quantitative analysis was determined in a standardless mode using ZAF (atomic number, absorbance, fluorescence) correction factors with the dedicated software (ESPRIT version 1.9, Bruker). Elemental distribution of all elements was investigated with X-ray EDS mapping employing 20 kV accelerating voltage, 105 μ A beam current, 5000X nominal magnification, and a $22 \times 25 \mu\text{m}$ collecting window.

Mechanical testing

All brackets from both groups were embedded in epoxy resin (Epofix, Struers, Belarup, Denmark), ground with water coolant SiC papers from 220 to 4000 grit, and polished with 3 and 1 μm diamond pastes (Struers) in a grinding/polishing machine (Ecomet III, Buelher, Lake Bluff, IL, USA). Then the specimens were ultrasonically cleaned for 10 min and left to dry. Instrumented Indentation Testing (IIT) measurements were carried out employing a universal hardness-testing machine ZHU0.2/Z2.5 (Zwick Roell, Ulm, Germany). Force indentation depth curves were monitored applying 9.8 N with 15

Figure 2: Representative backscattered electron image (BEI) from the brackets tested.

The rectangle indicates the region on the wing of brackets of X-ray collecting window (nominal magnification: 18X, bar: 2 mm).

sec dwell time by a Vickers indenter. Three readings were taken for each bracket and the mean value was used as representative of the specimen. All force-indentation depth curves were recorded and the Martens hardness (H_M), indentation modulus (E_{IT}), and elastic index (η_{IT}) were calculated according to ISO 14577-1 specifications [9]. After unloading, the Vickers hardness (HV) was determined based on the diagonal of the indentation.

Statistical analysis

The results of elemental content, H_M , E_{IT} , η_{IT} , and HV were statistically analyzed between the two groups by unpaired t-test at a 95% level of significance ($\alpha=0.05$).

Results

SEM/EDS analysis

Figure 3 shows a representative backscattered electron image (BEI) from the surfaces of both the tested groups. Both images show surface scratches with random orientation and a diffuse distribution of surface pores or fissures.

A second phase with higher mean atomic number (brighter areas) was also identified.

The quantitative results after EDX analysis are presented in Table 1. No significant differences before and after intraoral aging were determined for all elements tested.

Figure 4 shows BEI and X-ray maps of all elements tested. Au, Cu, and Ag shows similar distribution matches to the low mean atomic number contrast of corresponding BEI. Contrary Ir depicts complimentary distribution to the aforementioned elements following the higher mean atomic number contrast phase (bright areas) in BEI. Pt and Zn show a uniform distribution, while Al and Si have random distributions with a few high intensity areas following the pores in BEI.

Mechanical characterization

A representative force-indentation depth curve is presented in Figure 5, while the quantitative results for H_M , E_{IT} , η_{IT} , and HV are presented in Table 2 without statistically significant differences before and after intraoral aging.

Figure 3: Representative backscattered electron image (BEI) from the surface of REF (A) and INV (B) groups (bar: 10 μ m).

Table 1: Mean values (and standard deviations) for as received (REF) and retrieved brackets (INV).

Group	Au	Cu	Pt	Ag	Zn	Al	Si	Ir
REF	68.5(1.3)	13.4(1.0)	7.4(0.8)	7.8(0.9)	1.2(0.1)	0.9(0.5)	0.5(0.4)	0.3(0.3)
INV	68.4(0.9)	13.5(0.6)	7.8(0.5)	7.4(0.5)	1.3(0.2)	0.9(0.4)	0.2(0.2)	0.6(0.3)

No significant differences were found between elements tested ($p>0.05$).

Figure 4: Backscattered electron image (BEI) and X-ray distribution of Au, Cu, Pt, Ag, Ir, Al, Si, and Zn. Au, Cu, and Ag illustrate similar distribution complementary to Ir. The latter matches with the heavier phase (bright areas) to corresponding BEI. Pt and Zn show a uniform distribution, while Al and Si show a random distribution (bar: 5 μm).

Figure 5: Representative force indentation depth curve for both groups tested.

Table 2: Mean values (and standard deviations) for selected mechanical properties employing Instrumented Indentation Testing.

Group	Martens hardness (HM) (N/mm ²)	Indentation modulus (E_{IT}) (GPa)	Elastic index (η_{IT}) (%)	Vickers hardness
REF	2378(89)	70.5(1.7)	38.7(2.4)	246(7)
INV	2395(88)	70.8(0.9)	39.5(1.6)	243(5)

No statistically significant differences were identified between the two groups for all mechanical properties tested ($p > 0.05$).

Discussion

According to the results of this study, the composition, the microstructure, and the selected mechanical properties of the Incognito™ lingual brackets did not seem to be altered after intraoral aging, and thus the null hypothesis must be rejected.

These brackets are made of a Au-Cu-Ag-Pt alloy with low Zn content and traces of Al, Si, and Ir according to EDX analysis (Table 1). Small differences in quantitative results from a previous report [17] can be explained by a possible change in the alloy itself by the manufacturer. The spectra of this study were acquired from the surface of bracket while the previous one were taken from the bulk, which is another possible source of differentiation due to segregation of alloying elements during solidification. BEI revealed the distribution of a second phase with higher mean atomic number compared to the matrix, in accordance with a recent study [17]. The matrix is composed predominately Au-Cu-Ag as revealed by X-ray mapping, a finding which is appended to the high dissolution of Cu and Ag in Au structure. The heavier phase consists mainly of Ir due to the minimal dissolution of Ir in Au (<0.1 wt) in room temperature [1].

Neither the possible elements of dealloying (Cu and Ag) [14] from Au alloys nor the rest alloying elements showed any compositional alterations after intraoral exposure. This finding might be associated with two possible explanations. The one is that the specific formulation is not prone to dealloying process. The other is that two years is considered a short time for the relatively slow development of the dealloying process [14]. Both scenarios imply that the period of almost 2 years of intraoral exposure, which is a typical time of orthodontic treatment, might be considered a safe functional time from this prospect. However, it should not be taken as a conclusive outcome that release of metallic ions is impossible for this product, as the friction of orthodontic wires at the slot might trigger wear phenomena and thus the release of wear debris in oral cavity. However, this requires further experimental analysis.

E_{IT} and HV were found in accordance to previous report [17]. Although HM has been preferred recently [16] as it is free of various complications involved in Vickers testing (indentation size effect, resolution of optical system, operator's perception, elastic recovery around indentation after load removal, and others) [12], Vickers hardness was also recorded in this study for comparison purposes with literature data of orthodontic brackets and wires. Incognito™ shows the lowest HV value (246HV) compared to all commercially available metallic brackets made of Ti (297HV) [8], Ti-6Al-4V (392HV) [8], and Stainless Steel SS(363HV) [17]. The lower HV value of Incognito™ increases further the mismatch with harder orthodontic archwires (390–397 Titanium Molybdenum alloy TMA, 300–430 Nickel-Titanium alloy Ni-Ti [4], and 528–601 of stainless steel alloys SS recently) [16]. The much harder archwires might introduce plastic deformation in the softer bracket slot compromising the activation of the archwire and increasing friction, facilitating the wear phenomenon. Another important clinical point that should be further tested is the galvanic coupling of the precious alloy with the different types of orthodontic wires to avoid the galvanic corrosion of metallic orthodontic devices intraorally.

Conclusions

Intraoral aging of around 2 years of the tested Incognito™ high gold orthodontic brackets has no statistically significant effect on their mechanical, microstructural, and elemental properties. For that reason it can be expected that the tested brackets show similar mechanical properties during the whole orthodontic treatment.

Conflict of interest statement: The authors confirm that they have no competing or financial interests in the products used.

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