Metallurgical Characterization of Orthodontic Brackets Produced by Metal Injection Molding (MIM)

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Abstract: The aim of this study was to investigate the bonding base surface morphology, alloy type, microstructure, and hardness of four types of orthodontic brackets produced by Metal Injection Molding technology (Discovery, Extremo, Freedom, and Topic). The bonding base morphology of the brackets was evaluated by scanning electron microscopy (SEM). Brackets from each manufacturer were embedded in epoxy resin, and after metallographic grinding, polishing and coating were analyzed by x-ray energy-dispersive spectroscopic (EDS) microanalysis to assess their elemental composition. Then, the brackets were subjected to metallographic etching to reveal their metallurgical structure. The same specimen surfaces were repolished and used for Vickers microhardness measurements. The results were statistically analyzed with one-way analysis of variance and Student-Newman-Keuls multiple comparison test at the 0.05 level of significance. The findings of SEM observations showed a great variability in the base morphology design among the brackets tested. The x-ray EDS analysis demonstrated that each bracket was manufactured from different ferrous or Co-based alloys. Metallographic analysis showed the presence of a large grain size for the Discovery, Freedom, and Topic brackets and a much finer grain size for the Extremo bracket. Vickers hardness showed great variations among the brackets (Topic: 287 \pm 16, Freedom: 248 \pm 13, Discovery: 214 \pm 12, and Extremo: 154 \pm 9). The results of this study showed that there are significant differences in the base morphology, composition, microstructure, and microhardness among the brackets tested, which may anticipate significant clinical implications. (Angle Orthod 2005;75:1024–1031.)

Key Words: MIM; Brackets; Hardness

INTRODUCTION

Metallic orthodontic brackets are predominantly fabricated by casting and milling techniques.¹ However, during the past few years, a new method has been adopted for manufacturing metallic orthodontic brackets, the Metal Injection Molding (MIM).^{1–3} The MIM method was discovered and developed in the United States in the early 1980s and is especially suitable for the production of small parts.²

In the MIM process,^{2–4} metal powders with particle sizes of a few microns are mixed with organic binders

(typically, wax, thermoplastic resins, and other materials), lubricants, and dispersants, until a homogeneous mixture is obtained. Injection of the feedstock is done using an injection molding machine similar to those used in the plastics industry. The injected parts, called "green parts," are formed into the desired geometry but at 17–22% oversize to compensate shrinkage after sintering.

The next procedure is the "debinding," which is used to remove at least 90% of the organic binder from green parts by heat, solvent, or both. The green parts have now been transformed into "brown parts," preserving the same size with a quite porous structure. The final stage of the MIM process is sintering, which is performed in a high-temperature furnace under vacuum or a controlled atmosphere. In this stage the residual binder is removed, and at the end of the process the parts have shrunk by 17–22%, reaching the precise desired dimensions because shrinkage is similar along the three axes. Nevertheless, in certain cases, secondary operations such as thermal or surface treatments are required. MIM products have tight tol-

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FIGURE 1. Schematic representation of the MIM process.

erances of up to $\pm 0.3\%$ of the desired dimensions and density values more than 97% of the theoretical density of the material.^{3,4} The sequence of MIM production method is schematically presented in Figure 1.

Among the currently available manufacturing processes, MIM is the least expensive mainly due to material savings during the production cycle because runners and sprues can be easily recycled and reused. Casting is the most expensive because it is estimated that 90% of the metal used is wasted in sprues and runners³ and 50% to 75% of the material used becomes scrap during machining.⁵ MIM is considered the most competitive technology for the production of large quantities of complex and intricate parts, whereas milling is economically beneficial only for geometrically simple parts. For products with complex geometry, precision casting is a competitive technology for the production of small and medium quantities; for large quantities, there are some restrictions concerning limitations in the automation process.⁴ MIM allows the use of any alloy for the production of orthodontic brackets, which is not always the case with the other processes.

Apart from the economic advantages, the production method may have serious implications in the clinical performance of orthodontic brackets. The use of new alloys for the production of MIM brackets with different mechanical properties may affect their mechanical performance under clinical conditions. As single-piece appliances, MIM brackets are expected to be free of the corrosion consequences¹ associated with the galvanic couple of brazing alloys with stainless steel (SS) (Figure 2). Despite the sufficient number of MIM brackets that are commercially available, there is no information regarding the structural characteristics and mechanical properties of these appliances.

Therefore, the aim of this study was to investigate the base morphology, elemental composition, microstructure, and hardness of four orthodontic brackets produced by the MIM process.



FIGURE 2. Secondary electron images from (a) a cross section of a bracket consisting of two components (base and wing) joined together with a brazing alloy (indicated by the two white arrows) and (b) a single-piece bracket (original magnification $29 \times$).

TABLE 1. Commercial Names, Lot Numbers, and Manufacturers of Brackets Tested

Brand Name	Lot Number	Manufacturer
Discovery	329847	Dentaurum, Ispringen, Germany
Extremo	F2I23S07	Leone, Firenze, Italy
Freedom	95554	ClassOne, Lubbock, Tex
Topic	351585	Dentaurum, Ispringen, Germany

MATERIALS AND METHODS

Four metallic orthodontic brackets from each of the brands listed in Table 1 were studied. The surface texture quality of the bonding base of one bracket from each company was observed under a scanning electron microscope (SEM) (Quanta 200, FEI, Hillsboro, Ore) operating at 25-kV accelerating voltage and 110- μ A beam current.

These same four brackets from each company were



FIGURE 3. Secondary electron images from the retention surfaces of the brackets tested: (a) Discovery, (b) Extremo, (c) Freedom, and (d) Topic (original magnification $100 \times$). Discovery and Freedom with a laser-structured base, Extremo with a simple foil mesh, and Freedom with a microetched surface. Topic shows the denser network, followed by Freedom, Discovery, and Extremo.



FIGURE 4. The x-ray EDS spectra obtained from the polished surfaces of orthodontic brackets Discovery, Extremo, Freedom, and Topic.

TABLE 2. Elemental Compositions (wt%) of Alloys as Determined by Energy-Dispersive Spectroscopic Analysis for All Materials Tested

Element	Discovery	Extremo	Freedom	Торіс
Fe	68.2 ± 0.9	67.2 ± 1.1	68.9 ± 1.4	0.2 ± 0.0
Cr	17.6 ± 0.6	17.5 ± 0.4	20.2 ± 0.0	$30.6~\pm~0.8$
Ni	10.6 ± 1.3	10.5 ± 0.1		
Мо	2.2 ± 0.6	2.0 ± 0.3	3.1 ± 1.0	4.9 ± 0.5
Mn	0.6 ± 0.2	1.5 ± 0.3	1.2 ± 0.1	0.1 ± 0.0
Si	0.5 ± 0.1	0.7 ± 0.2	0.5 ± 0.5	1.4 ± 0.1
Cu		0.5 ± 0.2	3.8 ± 0.4	
Co			2.3 ± 0.2	62.5 ± 1.3

embedded in epoxy resin, ground with water coolant SiC papers from 220 to 2000 grit, and polished up to 0.05 μ m alumina slurry (Bueler, Lake Bluff, III) in a grinding/polishing machine (Ecomet III, Bueler). Specimens were cleaned in an ultrasonic bath for five minutes and vacuum coated with a thin layer of conductive carbon. The surface of cross sections were studied under the SEM, and their elemental composition was determined by energy-dispersive x-ray microanalysis using a Si(Li) energy-dispersive spectroscopic (EDS) detector (Sapphire, EDAX, Mahwah, NJ) with super

ultrathin window (Be). The x-ray EDS spectra were collected from each bracket under 25-kV accelerating voltage and 110- μ A beam current using an area analysis mode at 160× magnification, a 800- × 800- μ m sampling window, and 150-second acquisition time. The quantitative analysis of the percentage of weight concentration was performed by Genesis software (version 3.5, EDAX) under a nonstandard analysis, using ZAF correction methods.

The embedded specimens were repolished, and an etching solution composed of one g picric acid, five



10 µm

FIGURE 5. Microstructure of the brackets tested. (a) Discovery, (b) Extremo, (c) Freedom, and (d) Topic (original magnification 200×).

mL HCl, and 100 mL ethanol was used to reveal the microstructure of Discovery, Extremo, and Freedom brackets. The Topic bracket was electrolytically etched in five mL HCl, 95 mL H_2O solution, applying three V for 15 seconds. The microstructure of all brackets was studied under a reflected light microscope (Eclipse ME 600, Nikon, Kogaku, Japan).

The same specimens were repolished, and the exposed surfaces were used for the assessment of Vickers hardness (HV_{200}), using a microhardness tester (HMV-2000, Shimandzu, Tokyo, Japan) applying a load of 200 g and 15-second contact time. One reading was taken from each specimen, and the results of the hardness testing were statistically analyzed with one-way analysis of variance and the multiple comparison Student-Newman-Keuls test at 95% confidence level.

RESULTS

The results of surface texture investigation (Figure 3) showed distinct differences in base morphology among the brackets tested. Topic showed the most dense mesh network, followed by Freedom, Discovery, and Extremo.

Figure 4 presents representative x-ray EDS spectra from the polished surfaces of all the brackets tested. The results of the qualitative and quantitative analysis are presented in Table 2. According to the x-ray EDS analysis, each bracket is manufactured from a different alloy.

The microstructure of the brackets tested is presented in Figure 5. Discovery, Freedom, and Topic demonstrated large grains, whereas Extremo showed a much smaller grain size. A rather uniform distribution



FIGURE 6. Vickers hardness values of the brackets tested (mean \pm SD). All mean values showed statistically significance differences among the brackets (P = .05).

of pores was found in all brackets, except Extremo, where only isolated pore regions were detected.

The results of Vickers hardness (VHN) measurements are shown in Figure 6. Topic showed the highest VHN value, followed by Freedom, Discovery, and Extremo. According to the statistical analysis, all VHN values demonstrated significant differences among the groups tested (P < .05).

DISCUSSION

Although all brackets tested are produced by MIM method, each brand demonstrated a completely different surface morphology. The base pads of Discovery (Figure 3a) and Topic (Figure 3d) are produced by using a laser technique,⁶ which results in the melting and evaporation of the metal formulating hole-shaped retentive features on the base. The surface pattern of Extremo (Figure 3b) resembles the standard system with the simple foil mesh pad, and the surface of Freedom (Figure 3c) demonstrates a microetched pattern surface. It is well documented that the surface pattern geometry has a significant effect on bond strength to enamel.^{7,8} According to a previous study, Discovery with the laser-structured retention base demonstrated almost double the bond strength compared with Minitrim (Dentaurum) with a simple foil mesh pad.9 However, no information is available for the bond strength of the other brackets included in this study.

The results of EDS analysis showed that each bracket is manufactured from a different alloy. The elemental composition of Discovery and Extremo corresponds to austenitic-type SS. Freedom is a Ni-free Ferrous alloy with addition of Co and increased concentrations of Cr and Cu. Finally, Topic consists of a Co-based alloy. The elemental composition of alloys has a serious implication in the biocompatibility, corrosion resistance, and ionic release of orthodontic appliances.^{10–13} Although brackets produced by MIM technology are actually single-piece appliances and thus free from the corrosion risk associated with the galvanic couple of brazing alloys with SS,¹⁴ the biocompatibility and corrosion resistance can be greatly different among alloys with similar elemental composition.

Previous studies¹⁵ have pointed out Ni release in vivo from the SS PH 17-4 allov used for the production of bracket wing region but not from the 316 SS alloy used for the production of the bracket base probably because of higher corrosion resistance of the latter. According to the results shown in Table 2, only the composition of Discovery falls within the range of 316 SS16 [(wt %): Cr, 16-18; Ni, 10-14; Mo, 2-3; Mn, 2 max; Si, 0.75 max], a finding also reported by previous studies,9 whereas the composition of Extremo cannot be categorized as a 316 SS because of the presence of Cu. Moreover, the elemental composition of Extremo and ClassOne brackets cannot be classified in the range of alloys 303, 304, 304L, 316, 316L, and PH 17-4 SS,^{1,16–19} which are extensively used for the production of metallic brackets, and thus their corrosion and biocompatibility properties should be further investigated.

All the brackets tested revealed porosity, which may be assigned to the shrinkage of the green parts during sintering. Although theoretically the MIM parts have a density of more than 97% of the nominal value, a large numbers of factors (alloy, powder type, debinding method, sintering heat rate, sintering hold time etc) may influence porosity development during the manufacturing process.^{20,21} An almost uniform pore distribution is a known defect in MIM parts; therefore, efforts should be undertaken to eliminate this type of porosity because it decreases the mechanical strength and corrosion properties of MIM products.²²

Under the experimental conditions of this study, Vickers indentations were made by using 200 g and 15-second contact time. This load produces small pyramidal size, giving the ability to avoid the adverse effect of porosity on hardness measurements because intact areas were used for Vickers indentations. Moreover, these experimental conditions are routinely used for the evaluation of hardness of orthodontic brackets.^{15,19} Topic demonstrated the greatest hardness probably because of being a Co-Cr alloy rather than a ferrous alloy. Another interesting finding is that although Discovery and Extremo have small differences in their elemental compositions (the latter contains 0.5 %wt Cu and almost 1 %wt higher Mn), they demonstrate significant differences in Vickers hardness. Perhaps, the Extremo brackets were subjected to thermal treatments after fabrication such as stress-relief annealing that reduce the remaining stresses from the manufacturing process and thus hardness.

The Vickers hardness of the brackets tested varied from 154 to 287 VHN, which is much lower than the hardness (400 VHN)¹⁹ reported for the wing components of conventional SS brackets. This difference may have significant effects on the wear phenomena encountered during the archwire activation into the bracket slot. The SS archwires demonstrate a hardness of 600 VHN,23 whereas the hardness of NiTi archwires range from 300 to 430 VHN.24 The mismatch in hardness should be minimized to avoid wear phenomena during orthodontic treatment. The clinical significance of the hardness findings may pertain to the fact that low-hardness wing components may complicate the force transfer characteristics from activated archwires to teeth because it may preclude full engagement of the wire to the slot wall and possible plastic deformation of the wing.25 Based on hardness findings of the present study, it seems that MIM brackets are more compatible with NiTi archwires regarding the decrease in the consequences of hardness mismatch.

CONCLUSIONS

The results of this study showed that significant differences exist in the morphology of retention pads, alloy type, structure, and composition among the MIM brackets tested, which may affect the clinical performance of orthodontic brackets. Although MIM is the most competitive technology for brackets manufacturing, extensive clinical and laboratory research is required to establish the advantages of this technique.

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