FRACTURE CHARACTERISTICS, HARDNESS, AND GRAIN SIZE OF FIVE POLYCRYSTALLINE ALUMINA ORTHODONTIC BRACKETS

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ABSTRACT

While polycrystalline alumina ceramic brackets have superior esthetics compared to stainless steel brackets, there is concern about fracture of these ceramic brackets during clinical manipulation and debonding. The purpose of this study was to investigate the fracture characteristics and related properties of five currently marketed alumina maxillary central incisor brackets (0.018 x 0.022 inch slot width). The following products were selected: Clarity (Unitek/3M), Allure NSB (GAC), Intrigue (Lancer), Contour (Class One) and MXi (TP). Five samples of each bracket were resin-mounted, and the bases were polished using diamond abrasives. Vickers indentations were made on the bases, using a 1 kg load, to measure the hardness and determine the fracture toughness. Three sample brackets of each brand were notched with a diamond disk and fractured to reveal the bulk microstructure. In addition, another sample of each brand was left in the as-received condition for examination of the bracket design. All specimens were observed under the optical microscope, and then coated with a gold-palladium film and observed with a scanning electron microscope. Values of Vickers hardness and grain size were compared using analysis of variance (ANOVA) and the Ryan-Einot-Gabriel-Welsch (REGW) multiple range test.

Accurate fracture toughness values could not be determined because intergranular fracture yielded cracks that were not the required straight lines for the injection-molded

Contour and MXi brackets, and gross chipping-out of grains (also intergranular fracture) occurred for the conventionally molded Clarity, Allure NSB, and Intrigue brackets. The approximate mean fracture toughness value of the Contour brackets (4.6 $MPa \cdot m^{1/2}$) was significantly greater than for the MXi brackets (3.6 $MPa \cdot m^{1/2}$). The approximate Vickers hardness of the Contour brackets (3230 kg/mm^2) was also significantly greater than that for the MXi brackets (2970 kg/mm^2), which was significantly greater than that for the Intrigue, Clarity, and Allure NSB brackets (ranging from 2500-2360 kg/mm^2). The grain size of the Intrigue brackets (11 μm) was significantly greater than that for the Allure NSB and Clarity brackets (both 8 μm), which was significantly greater than that the MXi and Contour brackets (both 0.6 μm). Results suggest that fracture resistance varies for commercially available alumina brackets.

Dedicated to Dr. Dale Wade and Jan Wade

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CHAPTER 1

INTRODUCTION

With increasing numbers of adults seeking orthodontic treatment, the orthodontic community had to find an appliance with superior esthetics to the conventional stainless steel appliances. Newman (1965) is credited with the introduction of the clear unfilled polycarbonate brackets. These plastic brackets, however, were prone to staining and material deformation. In 1987, at the annual session of the American Association of Orthodontists in Montreal, the search for an effective esthetic bracket led to the introduction of the crystalline aluminum oxide ceramic bracket (Bramble, 1988). These ceramic alumina brackets were either very close to the natural shade of teeth or were transparent. Unlike the polycarbonate brackets, alumina brackets do not have problems with deformation or staining (Rains *et al.*, 1977; Aird and Durning, 1987). However, their clinical disadvantages include enamel wear, increased slot friction, tie-wing fractures during treatment, and excessive bond strength and fracture during debonding (Swartz, 1988; Douglass, 1989). Recently, the continuing quest for a clinically effective ceramic bracket has led to many innovations aiming at overcoming those aforementioned disadvantages. This project strives to describe these various innovations, and to elucidate certain physical and mechanical properties of polycrystalline aluminum oxide brackets.

Fracture Resistance of Alumina Brackets and Related Mechanical Properties

Aluminum oxide is well-known for its resistance to high temperature and chemical degradation, and for being one of the hardest natural materials. However, its resistance to fracture is low compared to metals such as stainless steel. According to Swartz (1988), ceramic materials such as alumina are brittle. In contrast to metals, which can be deformed considerably without fracturing, alumina exhibits essentially no plastic deformation prior to fracture. This is due to the fact that the strong covalent and ionic bonds in this material do not permit the movement of dislocations that occurs in metals. With alumina, when stresses reach critical levels, the interatomic bonds break and brittle failure occurs.

One of the mechanical properties which distinguishes ceramics from metals is fracture toughness. Scott (1988) stated that, although the hardness and the tensile strength of ceramic brackets substantially exceed that of metal brackets, the alumina brackets have much lower fracture toughness values (Fig. 1). Fracture toughness (K_{Ic}) is a measure of the strain-energy absorbing ability prior to fracture for a brittle material. This mechanical property is related to the level of tensile stress that must be exceeded at the tip of a crack before propagation results in material failure. Therefore, the fracture resistance of an alumina bracket depends on its fracture toughness. The higher the fracture toughness, the more difficult it is to propagate a crack in the material and thus the higher the fracture resistance (Eliades *et al.*, 1999). It has been reported that K_{Ic} for surgical grade polycrystalline alumina and stainless steel is in the range of 5-6 $MPa{\cdot}m^{1/2}$ and 80-95 $MPa{\cdot}m^{1/2}$, respectively (Scott, 1988; Christel *et al.*, 1989).

Fracture toughness is a useful measurement when comparing the fracture resistance of different orthodontic bracket materials, because it is a fundamental material property (Rosenstiel and Porter, 1988) and is not sensitive to bracket design. To determine fracture toughness values for ceramic materials, there are a variety of testing methods available, including the double torsion method (Goldman, 1985), the short rod with chevron notch method (Pilliar *et al.*, 1986), the single-edge-notched beam method (Lloyd and Adamson, 1987); the compact tension method (Kovarik *et al.*, 1991); and the indentation microfracture method (Ferracane, 1989). Although fracture toughness is independent of the mode of measurement, the first four mentioned testing methods require a larger specimen size and more specific specimen geometry (Fig. 2), while the indentation microfracture (IM) method can be applied to a smaller-sized specimen and to any available flat surface (*e.g.*, the polished base of a bracket) (Fig 3). Rosenstiel and Porter (1988) successfully used the indentation microfracture method to determine fracture toughness for dental ceramics. Smart *et al.* (1992), using the IM method, reported values of K_{Ic} for three commercial alumina brackets (2.7-5.2 $MPa \cdot m^{1/2}$). However, Butler (1992) found that the IM method was not suitable for testing the fracture toughness of alumina brackets, because of the intergranular mode of crack propagation.

Another measurement, which is related to fracture resistance and to fracture toughness, is the Vickers hardness. This indentation hardness is obtained when performing the Vickers IM technique to determine fracture toughness (Rosenstiel and Porter, 1988).

Fracture resistance is sensitive to internal flaws as well as surface flaws. Kusy (1988), who studied fracture toughness and tensile strength in relation to the surface morphology of a typical alumina bracket, believed that strength could be related to fracture toughness by a mathematical formula; the surface roughness was directly related to the flaw size for crack propagation. Swartz (1988) concluded that alumina ceramics are susceptible to crack propagation from minute external scratches, internal porosities and impurities, or sharp angles in bracket geometry; all of which result in local concentration of stresses. Therefore, the fracture resistance of a bracket is sensitive to the presence of local concentration of stresses.

The presence of an undesired local concentration of stresses can be mitigated through the manufacturing process. The actual fracture toughness of a bracket will be dependent on the manufacturing process, which can result in material inhomogeneity, porosity, and variation in both grain size and grain boundary quality (Morena *et al.*, 1986; Rosenstiel and Porter, 1988; Taira *et al.*, 1990; Tilson, 1994).

Polycrystalline alumina brackets are manufactured by mixing aluminum oxide particles with a binder so that the mixture can be molded into the shape of a bracket, which is then heated to temperatures in excess of 1800°C to burn out the binder and cause the particles to become joined as grains by a process known as sintering. Diamond cutting tools are then used to machine the slot dimensions. Heat treatment (annealing) is subsequently performed to relieve stresses caused by the cutting and to remove surface imperfections resulting from the manufacturing processes. While the average starting particle size is reported to be about 0.3 μm, the conventional bracket manufacturing process yields grains of 20-30 μm dimensions. The larger the ceramic grains, the greater the translucency, but when the grains reach a size of about 30μm, the material tends to become weaker (Kirchner and Gruver, 1970; Swartz, 1988). According to Kusy (1988), one of the remedies to improve fracture resistance is to decrease the grain size. Therefore, post-machining annealing must be carefully controlled to prevent further grain growth that would degrade the physical properties. Furthermore, the overall manufacturing process results in both structural imperfections at grain boundaries and the incorporation of trace amounts of impurities. These slight imperfections and impurities, even at levels of 0.001%, can serve as foci for crack propagation under stress (Swartz, 1988).

An alternative manufacturing process for polycrystalline alumina brackets is injection molding. This process does not require the brackets to be machined, and thus eliminates structural imperfections created by the cutting process; the fracture resistance of the injection-molded brackets is also reported to be improved (Bordeaux *et al.*, 1994).

Besides the method of manufacture, the geometry of the ceramic bracket is also critical for its fracture resistance. Finite element analysis (Fig. 4), which uses a computer model, has been employed to determine the stress level and stress distribution that will occur under loading and to design ceramic brackets that better accommodate the poor fracture resistance of alumina. Iwamoto *et al.* (1988) and Hansen (1997) found that greater stress was present at the deepest point of the ligature-tying slot and at the base of the wire slot. Therefore, the design of a fracture-resistant bracket must strive to eliminate stress concentrators, such as sharp angles from these slots.

Frictional and Debonding Properties of Alumina Brackets

The manufacturing process and design can also affect the frictional properties of alumina brackets. Since the bracket slot surface is relatively rough and since aluminum oxide is much harder than stainless steel, frictional resistance is greater during orthodontic sliding mechanics, and the alumina bracket can actually cause notching of the archwire (Angolkar *et al.*, 1990; Kusy and Whitley, 1990; Pratten *et al.*,1990; Tanne *et al.*, 1991; Alexander, 1992). In an *in vitro* study Omana *et al.* (1992) found that, for ceramic brackets, sharper slot edges could dig into the softer archwire material and increase friction, while smooth slot surfaces lowered sliding friction. They observed that injection-molded brackets (Contour – Class One Orthodontics; Ceramaflex – TP Orthodontics), with smoother slot surfaces and contours that were more rounded, appeared to create less friction than conventional ceramic brackets and to have frictional forces approaching those associated with metal brackets.

Aside from frictional properties, difficulty in debonding was also a problem with the early alumina bracket designs. One disadvantage of these brackets was fracture of the appliance during debonding. Stainless steel brackets, because of their ductility and lower elastic modulus, can be deformed in tension without fracture during debonding; deformation of the bracket during debonding initiates cracks in the adhesive layer. Alumina brackets, on the other hand, not only are more stiff (higher elastic modulus) and brittle than the stainless steel brackets, but also are designed with thicker sections to compensate for their lower fracture resistance. The tendency of alumina brackets to fracture during debonding is a consequence of their brittle nature and inability to undergo substantial deformation (Odegaard and Segner, 1988; Dischinger, 1990; Storm, 1990).

Excessive bond strength during debonding was also a concern with the early alumina brackets. Since aluminum oxide is an inert material, it cannot chemically bond to resin adhesives. The use of a silane coupled with an intermediate glass phase-containing layer on the bracket base allows chemical bonding to the resin adhesive. However, a chemically-retained ceramic bracket has higher bond strength when compared with metal brackets (Odegaard and Segner, 1988; Joseph and Rossouw, 1990; Bishara and Trulove, 1990; Winchester, 1991). During debonding, stronger bonding at the bracket-adhesive interface results in bond failure at the enamel-adhesive interface, which may lead to enamel fracture.

To overcome these problems with debonding, some manufacturers incorporated special designs into their brackets. The 3M Unitek company designed the Clarity bracket to collapse during debonding and initiate cracks in the adhesive. Bishara *et al.* (1997) found that these brackets could be debonded using forces similar to those required to debond metal brackets. The 3M Unitek company and some other manufacturers have also used mechanical interlocking as an alternative to chemical retention. Guess *et al.* (1988) suggested that mechanical retention would provide adequate orthodontic bond strength. Protruding crystals, recesses, and grooves provide mechanical interlocking with the resin adhesives.

The TP Orthodontics company has added a cast epoxy mesh base to their MXi bracket to facilitate the debonding process. According to Devanathan (1997), this epoxy base, which is chemically bonded to the bracket, would adhere to most orthodontic adhesives, and deform and initiate cracks in the adhesive during debonding. Bishara *et al.* (1999) found that the MXi brackets had a lower shear bond strength than the Clarity brackets, but still higher than the minimal clinically acceptable level of 6-8 MPa suggested by Reynolds (1975).

Other bracket base designs employ a combination of chemical and mechanical retention. The GAC company incorporates silane-coated and recessed dimples in their Allure brackets, while Lancer uses silane-coated grooves in their Intrigue brackets. This dual approach for reducing alumina bracket retention decreased the bond strength sufficiently to prevent enamel damage, while maintaining clinically adequate levels (Bordeaux *et al.*, 1994).

Because of the clinical importance of the fracture behavior of alumina brackets, it was considered worthwhile to investigate this behavior for several recently marketed brands of these brackets. In addition, there was controversy between the results of Smart *et al.* (1992), who reported use of the Vickers MI technique to determine the fracture toughness of two brands of alumina brackets, and Butler (1992), who found that this technique could not be employed for alumina brackets.

This study has attempted to use the Vickers MI technique to determine the fracture toughness and the Vickers hardness (K_{Ic}) for five brands of polycrystalline alumina brackets (Clarity, 3M Unitek; Allure NSB, GAC; Contour, Class One Orthodontics; Intrigue, Lancer; MXi, TP Orthodontics). The mean grain size for each of these brands was also determined by fracturing the brackets and analyzing their fracture surfaces. Two of the brands (Contour and MXi) were manufactured by a recently introduced injection-molding process. The Clarity, Allure NSB and Intrigue brackets were manufactured by conventional processing methods similar to those used for the polycrystalline alumina brackets investigated by Butler (1992).

The Vickers hardness of these brackets was measured, and K_{Ic} values were calculated (where possible), using an equation relating the fracture toughness to the

Vickers hardness (Rosenstiel and Porter, 1988). A modified intercept method was used to determine the mean grain size (Van Vlack, 1989) (Fig 5). The scanning electron microscope was used to determine the fracture behavior and microstructure of brackets that were indented with the Vickers hardness tester and of brackets halves that had been cleaved with a chisel.

Null Hypotheses for Research

It is intended that the information obtained from this research will help the orthodontist to be more informed regarding the relative fracture resistance of different alumina brackets and the rational selection of products based on their desired properties. Therefore, this study intends to disprove the following null hypotheses (H_o):

1. The indentation microfracture method is not a viable method to determine the fracture toughness of polycrystalline alumina brackets.
2. There is no difference in grain size among the different brands of polycrystalline alumina brackets.
3. There is no difference in surface smoothness among the different brands of polycrystalline alumina brackets.
4. There is no difference in the fracture resistance among different brands of polycrystalline alumina brackets.

CHAPTER 2

MATERIALS AND METHODS

Materials

Ten polycrystalline alumina orthodontic brackets were donated by each of the following companies: 3M Unitek, Monrovia, California (Clarity); GAC, Central Islip, New York (Allure NSB); Lancer, San Marcos, California (Intrigue); Class One Orthodontics, Lubbock, Texas (Contour); TP Orthodontics, LaPorte, Indiana (MXi). The Clarity, Allure NSB, and Intrigue brackets were manufactured using the conventional molding/sintering/machining method described in Chapter I. The Contour and MXi brackets were produced by the injection molding method. All brackets selected for this study were designed for use with maxillary central incisors (either right or left), because these are the largest and least convex of all brackets made. All brackets had 0.018 x 0.022 inch slot dimensions.

Methods

Attempts were made to determined fracture toughness (K_{Ic}) of the alumina brackets by the indentation microfracture method described in Chapter I, in which K_{Ic} is calculated from the average length of the four radial cracks that emanate from the corners of a Vickers microhardness indentation. The equation used to calculate fracture toughness is as follows (Anstis *et al.*, 1981; Rosenstiel and Porter, 1988):

$$K_{Ic} = 0.016\ (E/H)^{1/2}\ (P/c_o^{3/2}).$$

In this equation P is the peak load, c_o is the crack length at equilibrium after the indenter is released (c_o is measured from the center of the Vickers hardness indentation to the tip of the crack), E is the elastic modulus of alumina (55 x 10^6 psi) (Kingery *et al.*, 1976), and H is the Vickers hardness (Rosenstiel and Porter, 1988).

In an initial pilot study, loads of 0.5 kg, 1 kg, 5 kg, and 10 kg were applied to one representative bracket sample of each brand to determine the optimal load. The objective was to achieve the largest indentation that had radial cracks approximately equal to half the length of the diagonal of the indentation and without any lateral chipping (Fig. 3). This length of the radial cracks is considered the optimum for determination of fracture toughness (Anstis *et al.*, 1981). From the pilot study, 1 kg was found to be the optimal load. Loads that were larger than 1 kg produced heavy damage to the bracket and rendered the indentation pattern unrecognizable, and a 0.5 kg load did not yield indentations that were visible at X100 magnification in the microscope associated with the hardness tester.

Five randomly selected brackets from each company were mounted in individual transparent epoxy resin disks (Leco, St. Joseph, Michigan). With the wings buried inside the epoxy and the bracket bases exposed, the bases were then polished with a series of abrasive papers (Struers, Westlake, Ohio), culminating in a diamond slurry of 0.05 μm abrasive particles. Each sample bracket was then subjected to a localized fracture process with the aid of the Vickers microhardness tester (Leco).

Using a 1 kg load, two indentations were placed on each polished bracket base. The samples (mounted in resin) were then vacuum sputter-coated with an approximately 20-nm thick gold-palladium conducting film. The coatings were necessary to prevent charging of the nonconducting alumina specimens from the electron beam of the scanning electron microscope (SEM) and to facilitate identification of the tips of the cracks after indenting. The microstructure of each bracket was observed, and representative regions and indentations were photographed in the secondary electron emission image mode of the SEM (JSM-820, JEOL Ltd, Tokyo, Japan). Vickers hardness values were calculated by using the equation:

$$VHN = 1.8544\ L/\ d^2$$

In this equation, L is the load in kg, and d is the mean diagonal length of the indentation pattern in mm. One-way analysis of variance (ANOVA), followed by the Ryan-Einot-Gabriel-Welsch (REGW) multiple range test, was performed to determine whether there were statistically significant differences among the mean hardness values for the five brands of brackets at the $\alpha = 0.05$ level.

Three additional sample brackets of each brand were then notched with a diamond disk and fractured with a chisel. These fractured bracket halves were also mounted and coated with a gold-palladium film. The fracture surface morphology of each bracket was observed, and representative SEM photomicrographs were taken. The mean grain sizes for the five polycrystalline brackets were calculated directly from the SEM photomicrographs using a modified intercept method (Van Vlack, 1989) (Fig. 5). The mean chord length (L) is an index of grain size. The value of L can be determined by placing a random circle of known circumference across the SEM photomicrographs of the *fracture surface*. The mean chord length (L) is the reciprocal of the number of boundary intersection points per unit length, P_L, where

$$L = 1/P_L$$

A 50 mm diameter circle was placed randomly on the photomicrograph of the fracture surface, and the number of grain boundaries it intersected was counted. At a magnification of X1000, the length of the circumference of the 50 mm diameter circle on the photomicrograph is equal to π (50mm/1000) = 0.16 mm or 160 μm. The value of P_L is equal to the number of intersections divided by 0.16 mm, and L can then be calculated from P_L.

SEM photomicrographs were also taken of a randomly selected representative (as-delivered) bracket of each brand to illustrate the various designs.

CHAPTER 3

RESULTS

Fracture Characteristics

As discussed in Chapter II, pilot experiments showed that 1 kg was the optimal load for producing recognizable indentation and radial crack patterns for all bracket brands. However, three (Clarity, Allure NSB, and Intrigue) out of the five brands displayed predominantly gross chipping out of grains (intergranular fracture) under the 1 kg loading imposed by the Vickers indenter. The grain boundaries were clearly seen on photomicrographs of these indented brackets. In addition, cleavage (transgranular fracture) across the alumina grains was apparent. The Clarity brackets showed the least chipping out of these three brands, while the Allure NSB and Intrigue brackets displayed similar chipping patterns (Figs. 6 and 7). Only the Contour and MXi brackets appeared to exhibit the straight line crack pattern desired for fracture toughness determination (Fig. 8).

However, at higher magnifications (X1500 – X2000), the crack propagation was also intergranular for the Contour and MXi brackets (Fig. 9). In addition, for two of the five brackets for each of the Contour and MXi bracket groups, the crack lengths were too long to be measured on the high magnification SEM screen. Measurement at low magnification was not possible, because the crack tips could not be seen. Therefore, mean Vickers hardness values were calculated from the measured diagonals of all five samples for each of the five brands, while approximate mean fracture toughness values were estimated for only the three remaining samples for each of the two injection-molded brands (Table 1). Based upon the ANOVA and the REGW multiple range tests, there was no significant difference (P > 0.05) in hardness among the three brands (Clarity, Allure NSB and Intrigue) in the conventional bracket group. However, there was a significant difference in Vickers hardness between the two brands in the injection-molded group (Contour and MXi), as well as between the two main groups of brackets (conventional vs. injection molded). Based on Student's t-test, the alumina in the Contour bracket and the alumina in the MXI bracket do not have significantly different values of fracture toughness (P > 0.05). However, this conclusion must be regarded as tentative, since fracture toughness values could not be obtained for two of the five Contour and MXi brackets. The much longer crack lengths in those brackets were indicative of considerably lower values of fracture toughness, presumably because of microstructural flaws associated with processing by the manufacturers.

SEM examination of the *surfaces of the as-delivered (not fractured) brackets* revealed that the two brands of injection-molded brackets had very smooth surfaces, compared to the three conventionally processed brackets. At X250 magnification, evidence of individual grains was not discernible on the surfaces of the injection-molded brackets (Figs. 10 and 11), whereas individual grain boundaries and plucked-out grains were evident on the surfaces of the conventionally processed brackets at the same magnification (Fig. 12). Observation of the *fracture surfaces of the brackets* revealed that the mean grain sizes for the conventionally processed brackets (Clarity, Allure NSB, and Intrigue) (Figs. 13 and 14) were over an order of magnitude larger than those for the two injection-molded brackets (Contour and MXi) (Fig. 15).

Based upon the ANOVA and the REGW multiple range tests, there were highly significant differences in grain size among the five brands of brackets (Table 2).

The conventionally processed Intrigue brackets had the largest grain size, while the injection-molded brackets (Contour and MXi) possessed the smallest grain size. There was no significant difference between the grain sizes of the conventionally processed Allure NSB and Clarity brackets, and there was no significant difference between the grain sizes of the two injection-molded brackets.

Designs

Regarding bracket designs, Clarity (3M Unitek) uses a metal insert to provide a smooth slot surface for archwire engagement (Fig. 16) and incorporates a stress-concentrating defect in the bracket base to improve debonding characteristics (Figs. 17 and 18). The MXi bracket (TP Orthodontics), on the other hand, uses a cast epoxy base to improve debonding characteristics (Fig. 19). Both the Clarity and Contour (Class One Orthodontics) brackets have protruding crystals for mechanical retention (Fig. 20), while both the Intrigue (Lancer) and Allure (GAC) brackets use recesses (Figs. 21 and 22).

The bracket geometry also varies among the different brands of brackets, as judged by qualitative SEM observations (Figs 23-26). The Contour and Clarity brackets have the lowest tie-wing profile (Figs. 23Aand B). The base of the tie-wings has the greatest bulk for the Clarity bracket (Fig. 23A) and the least bulk for the MXi bracket

(Fig. 23D). The Allure and Intrigue brackets possess sharper edges in the milled archwire slot (Figs. 24 and 25) than the rest of the brackets; the edges of the archwire slots for the Contour bracket appeared to have the greatest extent of rounding (Fig. 26). The archwire slot depths were greater (approximately 0.75 mm) for the Allure, Intrigue, and MXi brackets than for the Clarity and Contour brackets (0.50 mm) (Figs. 23A-E). None of the brackets in this study presented any sharp projections.

CHAPTER 4

DISCUSSION

Fracture Resistance

The indentation technique, previously used by Rosenstiel and Porter (1988) to evaluate fracture toughness of dental ceramics containing a glass phase, was found to not be suitable for the polycrystalline alumina brackets evaluated in this study, because the required straight radial cracks for calculation of K_{Ic} could not be produced. Therefore, the formula for K_{Ic}, which was described by Rosenstiel and Porter (1988), Kusy (1988), and Scott (1988), could not be used to calculate the true fracture toughness of the alumina brackets. Therefore the first null hypothesis for this study was accepted, namely that the Vickers IM method is not a suitable technique for determining the fracture toughness of alumina orthodontic brackets. The present research confirms the previous conclusion by Butler (1992), who also found that the Vickers IM method was not suitable for determination of fracture toughness of the conventionally processed ceramic brackets marketed at that time.

With the polycrystalline alumina brackets evaluated in this study, in agreement with the observations of Butler (1992), the fracture predominantly followed complicated paths along grain boundaries rather than across cleavage planes within the grains; though, some intragranular fracture did also occur. Apparently, with this ceramic material, well-defined straight radial cracks emanating from the corners of the indentation cannot be achieved. This result is plausible since, according to the principles of material science, the path of crack propagation in polycrystalline alumina is expected to follow the grain boundaries, rather than the much more difficult paths across grains (transgranular fracture) (Heuer, 1969; Johari, 1974; Kingery *et al.*, 1976; Eliades *et al.*, 1994; Eliades *et al.*, to be published). Therefore, the estimated fracture toughness values for the Contour and MXi brackets are not accurate (3.6 and 4.6 $MPa \cdot m^{1/2}$ respectively), although they are comparable to the values reported by Smart *et al* (1992) (2.7-5.2 $MPa \cdot m^{1/2}$), and approach those reported for (very high-quality) surgical-grade alumina (5-6 $MPa \cdot m^{1/2}$) (Christel *et al.*, 1989.

Another problem encountered with the attempted measurement of fracture of the polycrystalline alumina brackets was the observation of much longer radial cracks in some indented brackets, implying much lower values of fracture toughness. Presumably, these much longer cracks at the tips of the Vickers indentations in two of the five samples for both the Contour and MXI brackets arose from microscopic porosity or other defects, arising from the bracket manufacturing process. It is also possible that these longer radial cracks were instead associated with more extensive subsurface fracture of the injection-molded brackets as a result of the large stresses arising from the indenter.

In order to obtain the true fracture toughness of the basic alumina comprising the brackets evaluated in this study, bulk alumina specimens much larger than orthodontic brackets must be obtained, and K_{Ic} would have to be determined by one of the other techniques listed in Chapter I.

Alternative experiments to measure the fracture resistance of entire brackets or tie-wings have been reported in the literature, *e.g.*, using a chisel to apply a shear load or a harness to apply a tensile load on the wings to fracture the brackets (Bordeaux *et al.*, 1994). However, such tests are highly sensitive to the bracket design and were not considered appropriate for the present investigation where the fracture behavior of the basic alumina materials were studied. Nevertheless, a future study, where loads to fracture entire brackets or tie-wings are determined, would provide useful practical information for the orthodontist who is concerned about the selection of ceramic brackets with the lowest resistance to fracture.

Although the Vickers indentation technique could not be used to calculate K_{Ic}, approximate Vickers hardness values could be determined for the alumina brackets. These hardness values cannot be regarded as exact (in the same manner that such measurements are considered for metals), because an unknown amount of the indenting energy was always expended on crack formation and propagation and the chipping out of grains. Nevertheless, Contour possessed the highest mean (approximate) hardness value and the two injection-molded alumina brackets were harder than their conventional molded/sintered/machined counterparts (Table 1). In fact, the hardness values for Contour and MXi (2970 and 3230 kg/mm^2) may exceed those reported for surgical grade alumina; the values of 2000-3000 kg/mm^2 reported by Christel *et al.* (1989) appear to also be approximate.

According to Kusy (1988), one of the remedies to improve the strength and fracture resistance of alumina orthodontic brackets is to decrease the grain size. The present SEM observations of fracture surfaces of the five brands of brackets, combined with the modified intercept method, revealed the smallest mean grain sizes (0.57 and 0.65 μm) occurred for the two injection-molded brackets, and the largest mean grain size

(11.33 μm) was found for the Intrigue brackets (Table 2). The mean grain sizes for the other two conventionally processed alumina brackets (Clarity and Allure NSB) were approximately 8 μm. Therefore the second null hypothesis for this study was rejected.

Considering the values of approximate Vickers hardness and mean grain size in Table 2, it is hypothesized that the two brands of injection-molded brackets might have higher resistance to fracture than the three brands of conventionally processed brackets. Since the tensile strength of a ceramic bracket is highly dependent on its surface condition, the injection-molded brackets (Fig. 12), which have a visually much smoother surface as observed with the SEM_than the conventionally processed brackets (Figs. 10 and 11), would be expected to possess higher resistance to fracture under tensile loading. The SEM observations clearly indicated that the origin of the surface roughness for these five brands of alumina brackets was the loss or pluck-out of individual alumina grains. Therefore the third null hypothesis for this study was rejected.

Although quantitative fracture toughness values could not be measured accurately in this study, some qualitative information about the relative fracture resistance and failure strength could still be determined from the extent of indentation destruction and the grain size, respectively. The injection-molded brackets appeared to have the highest fracture resistance, qualitatively, because they exhibited no gross chipping with indentation and they are composed of the smallest grains. Of the three conventionally processed brackets, the Clarity brackets exhibited the least amount of gross chipping from the indentation loading. Therefore, the present qualitative SEM examination of the five brands of polycrystalline alumina brackets suggest that the ranking for relative fracture resistance should be as follows: Contour (Class One Orthodontics) and MXi (TP

Orthodontics) [highest]; Clarity (3M Unitek) [intermediate]; Allure NSB (GAC) and Intrigue (Lancer) [lowest]. Therefore the fourth null hypothesis for this study is rejected, although a future study of the fracture behavior of the tie-wings or the entire brackets under clinically realistic conditions needs to be performed to validate this conclusion.

Besides the mechanical and physical properties of the ceramic brackets, their design and surface finish also will contribute to their fracture resistance. As discussed in Chapter I, finite element analysis has been used to describe the stress distribution under loading and to design ceramic brackets that accommodate the poor fracture resistance of alumina. Iwamoto *et al.* (1988) and Hansen (1997) found that greater stress was present at the deepest point of the ligature-tying slot and at the base of the wire slot. Thus, it is not unexpected that four out of the five manufacturers (Class One Orthodontics, 3M Unitek, Lancer, and GAC) incorporated a thicker tie-wing base design to resist both the compressive and tensile stresses expected during clinical manipulation and debonding. The Contour and Clarity brackets adopted the lower-profile tie-wing design to reduce the lever-arm effect that occurs when perpendicular loads are applied to the tie-wings, thereby reducing the tensile stresses that are expected under clinical conditions. When considering the guidelines suggested by Kusy (1988) to minimize the occurrence of brittle failure arising from surface imperfections and sharp intersections, only the Contour bracket had the recommended smooth surface finish and rounded intersections. Although the Clarity and Intrigue brackets did not possess the smooth surface of an injection-molded bracket, their designs also provided smooth intersections. The Allure NSB bracket, except for the machined archwire slot, also had round intersections; however, this milled slot might serve as a stress concentrator under torquing loads.

According to Hansen (1997), to achieve reduction of tensile stresses under loading and improve torquing strength, 3M Unitek intentionally placed the Clarity bracket in a state of compression, a technique used in the construction industry to reinforce concrete. This was achieved through the metal insert; the metal and ceramic were joined at high temperature. Since the shrinkage rate on cooling was twice as great for stainless steel as for alumina, the difference in thermal contractions generated a compressive stress field in the ceramic as the bracket cooled. Tensile loads applied to this bracket must overcome the prestressed compressive loads before tensile stresses can appear and cause fracture. It was also suggested by Hansen (1997) that the metal insert helped strengthen the bracket to withstand orthodontic torquing forces. In addition, with the advent of the resilient nickel-titanium and beta-titanium archwires, which are able to deliver constant and gentle forces, torquing loads may be decreased. Therefore, state-of-the-art archwires combined with better bracket designs and improved manufacturing process will help to improve the fracture resistance of ceramic alumina brackets.

Frictional and Debonding Properties

Archwire-bracket sliding friction is still a major concern for alumina orthodontic brackets. The metal slot insert of the Clarity bracket also appears to have helped solve the frictional problem of the alumina bracket. As described in Chapter I, Omana *et al.* (1992) found that, for ceramic brackets, the sharp and hard slot edges could dig into the softer archwire material and increase friction. They also concluded that the Contour and Ceramaflex injection-molded brackets, which have smooth surfaces and round contours, appear to create less friction than the conventionally processed ceramic brackets. In their study, SEM photographs confirmed that the Contour and MXi brackets possessed smoother slot surface and rounder slot edges than the Allure and Intrigue brackets. According to Omana *et al.* (1992), the frictional properties of the injection-molded brackets approached those of stainless steel brackets. However, since this was an *in vitro* study, it would be interesting to establish through long-term *in vivo* studies whether the injection-molded brackets would also cause frictional wear on the archwires.

Another major orthodontic concern is the debonding characteristics of ceramic brackets. Stainless steel brackets, because of their ductility and lower elastic modulus, can be deformed under tensile stresses without fracture during debonding. It was noted in Chapter I that this bracket deformation initiates cracks in the adhesive layer to facilitate debonding. Ceramic brackets on the other hand, are not only more stiff and brittle, they are designed with thicker sections to compensate for their lower fracture resistance. Therefore, ceramic brackets are more difficult to debond, since they are not easily deformed. To overcome this problem, some manufacturers have incorporated special designs into their brackets. SEM examination confirmed that 3M Unitek had incorporated a local stress concentrator into the base of the Clarity bracket (Figs. 17 and 18), which would allow the bracket to collapse and initiate cracks in the adhesive during debonding.

SEM photographs showed that TP Orthodontics had added a cast epoxy mesh base to the MXi bracket to help ease the debonding process (Fig. 19). Bishara *et al.* (1999) found that these brackets had a lower shear bond strength than the Clarity brackets, but still higher than the minimal, clinically acceptable level of 6-8 MPa suggested by Reynolds (1975). SEM examination also showed that the Allure NSB and

Intrigue brackets employed silane-coated recessed dimples and grooves, respectively, to lower the bond strength sufficiently to prevent enamel damage, but still remain clinically adequate (Bordeaux *et al.*, 1994).

CHAPTER 5

SUMMARY AND CONCLUSIONS

The Vickers indentation microfracture (IM) method is not suitable in determining the fracture toughness of polycrystalline alumina brackets. Because well-defined straight radial cracks emanating from the corners of the indentation cannot be achieved, the polycrystalline brackets are not amenable to the use of the IM method to calculate fracture toughness. However, approximate Vickers hardness values could be measured, which indicated that the alumina in the Contour brackets is the hardest, followed by MXi, and then the conventionally processed brackets.

There are substantial differences in the grain size and surface smoothness among the five brands of alumina brackets evaluated in this study. The injection-molded Contour and MXi brackets have the smaller grain size and smoother surface, whereas the three conventionally processed alumina brackets had grain sizes an order of magnitude greater. The surface roughness of all of these ceramic brackets is considered to arise from pluck-out of the alumina grains and therefore correlates with the grain size.

Fracture resistance also appears to vary among the five different brands of alumina brackets. Although physical and mechanical properties suggest that the injection-molded brackets possess higher fracture resistance than the conventional brackets, each of the three conventional-molded-bracket manufacturers have improved the overall fracture resistance of their appliances through various designs. An additional investigation would be required to measure the fracture resistance of the brackets directly, but the values of fracture load or stress would be strongly influenced by differences in the bracket designs. Regarding the frictional and debonding properties of alumina brackets, the present SEM observations suggest that Lancer and GAC have incorporated designs into their bracket bases to facilitate debonding, while 3M Unitek, TP Orthodontics, and Class One Orthodontics have provided design features for their brackets that address both of these major clinical concerns.

APPENDIX A

TABLES

Bracket	Vickers Hardness Values* (mean ± SD in units of 10^3 kg/mm^2)	Fracture Toughness** (estimated mean ± SD in units of MPa·m$^{1/2}$)
Clarity (3M Unitek)	2.44 ± 0.24 **C**	Could not be calculated.
Allure NSB (GAC)	2.36 ± 0.16 **C**	Could not be calculated.
Intrigue (Lancer)	2.50 ± 0.11 **C**	Could not be calculated.
Contour (Class One Orthodontics)	3.23 ± 0.17 **A**	4.57 ± 0.85
MXi (TP Orthodontics)	2.97 ± 0.06 **B**	3.58 ± 0.23

*Obtained from five specimens for each brand. Mean values with the same REGW letter code were not significantly different ($P > 0.05$).
**Obtained from three specimens for each brand. Using Student's t test, mean values for the Contour and MXi brackets were not significantly different.

Table 1. Mean Vickers Hardness and Estimated Fracture Toughness Values

Bracket	Grain Size* (mean ± SD in microns)
Clarity (3M Unitek)	7.72 ± 0.40 **B**
Allure NSB (GAC)	8.22 ± 1.02 **B**
Intrigue (Lancer)	11.33 ± 2.15 **A**
Contour (Class One Orthodontics)	0.57 ± 0.05 **C**
MXi (TP Orthodontics)	0.65 ± 0.08 **C**

*Mean values with the same REGW letter code were not significantly different ($P > 0.05$). Measurements were performed on three sample brackets of each brand.

Table 2. Mean Grain Sizes According to the Modified Intercept Method

APPENDIX B

FIGURES

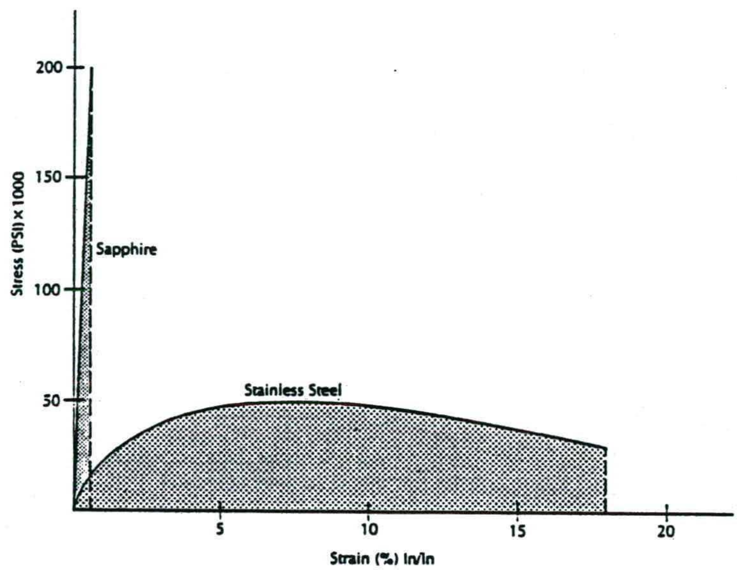


Figure 1. Stress-strain curves for sapphire (single-crystal aluminum oxide) and stainless steel (Scott, 1988).

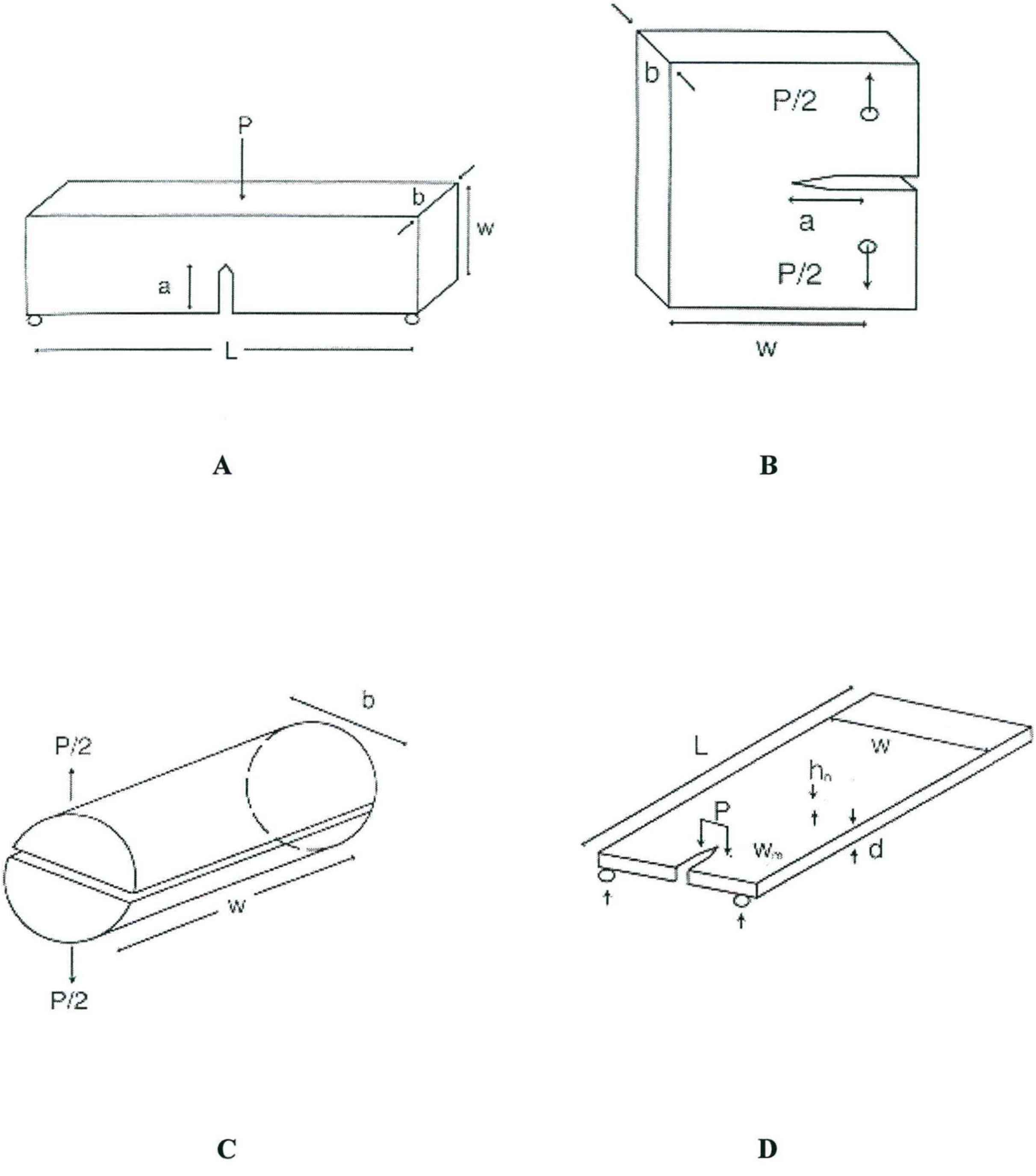
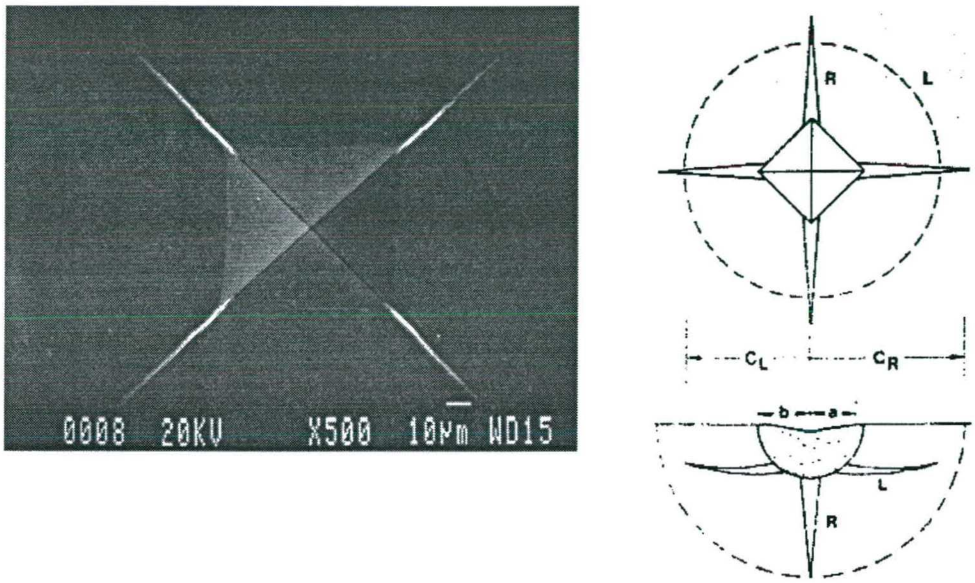


Figure 2. Specimen geometries for the determination of fracture toughness: Single-edge notched method (A) (Lloyd and Adamson, 1987); compact tension method (B) (Kovarik *et al.,1991*); short rod with chevron notch method (C) (Pilliar *et al., 1986);* double torsion method (D) (Goldman 1985).



A **B**

Figure 3. SEM photograph of a Vickers indentation on a zirconia orthodontic bracket (A) (Tilson, 1994; (B) top and side-view diagrams of an ideal indentation pattern, where a + b represents the diagonal length and C_L and C_R represent the left and right crack lengths (Morena *et al.*, 1986).

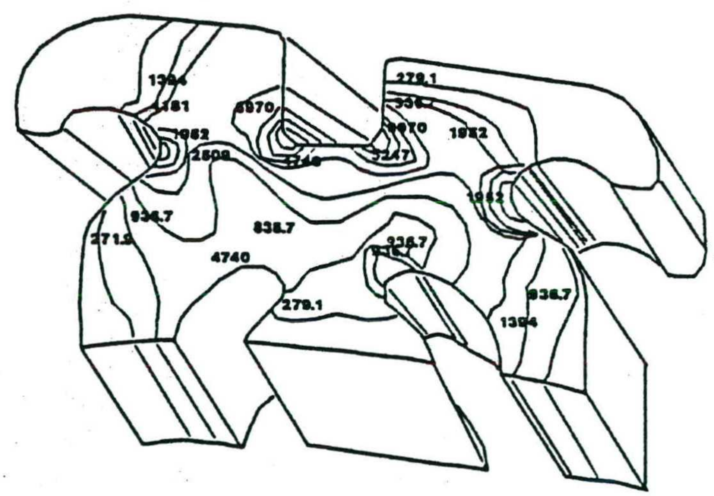


Figure 4. Finite element analysis showing stress distribution in a bracket subjected to loading (Swartz, 1988).

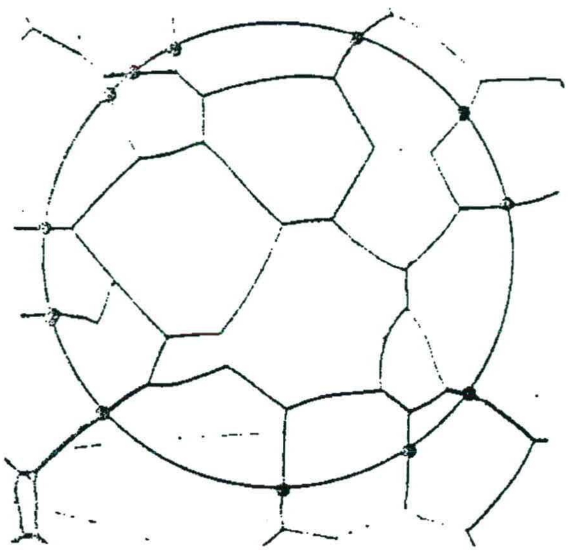


Figure 5. A modified intercept method diagram showing grain boundary intercepts on a 50 mm diameter circle (Van Vlack, 1989).

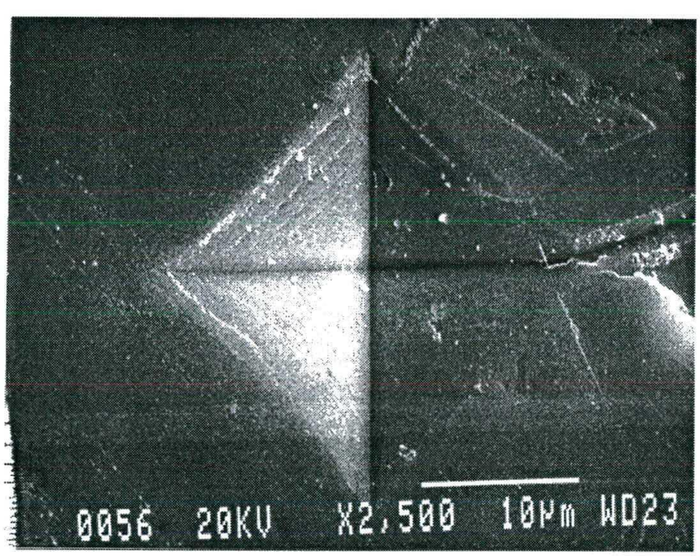


Figure 6. Scanning electron micrograph of a 1 kg indentation on the polished base of a Clarity bracket.

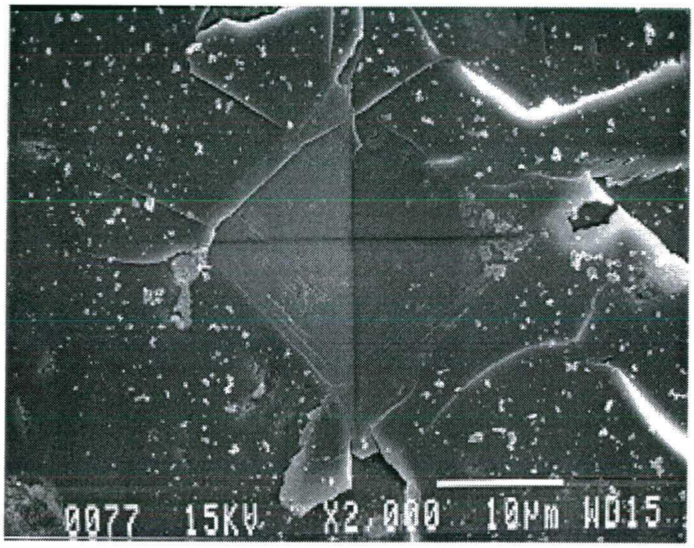


Figure 7. A 1 kg indentation on the polished base of an Allure bracket.

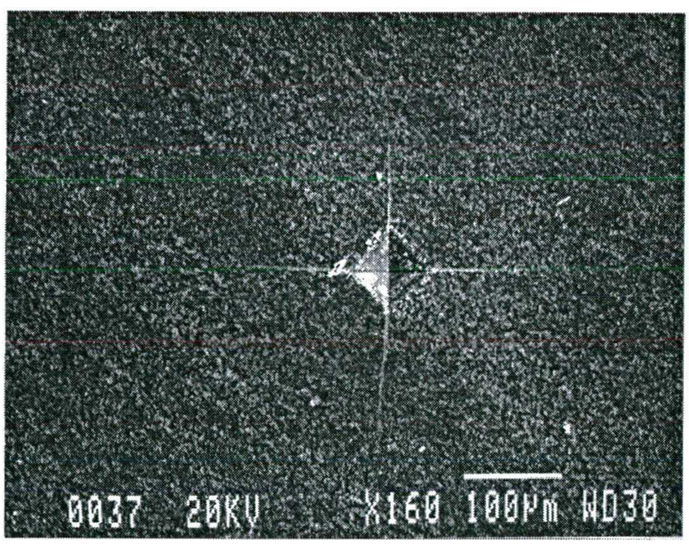


Figure 8. A 1 kg indentation on the polished base of a Contour bracket (low magnification).

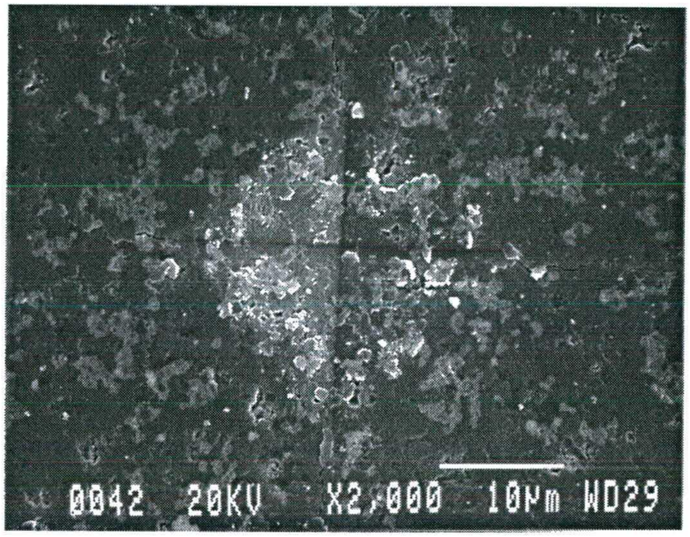


Figure 9. A 1 kg indentation on the polished base of an MXi bracket (high magnification).

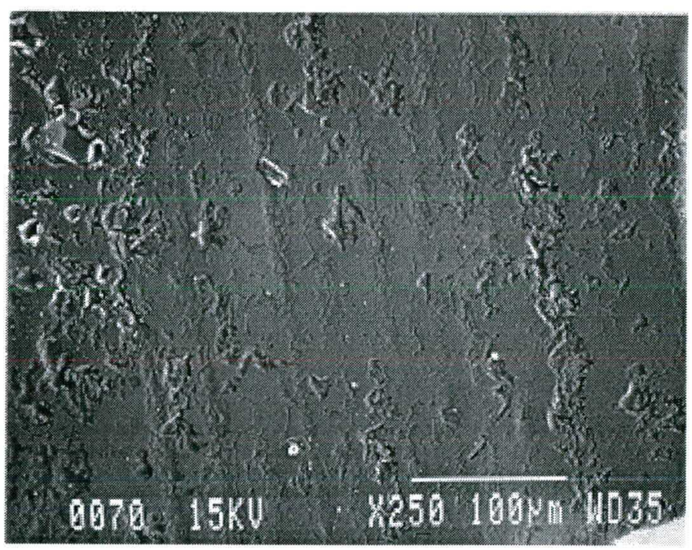


Figure 10. SEM photograph of the surface of an Allure NSB bracket (*as received*).

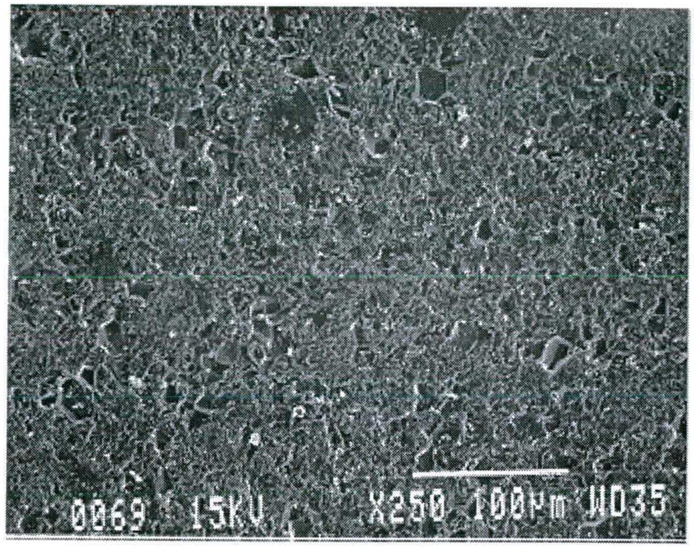


Figure 11. SEM photograph of the surface of a Clarity bracket (*as received*).

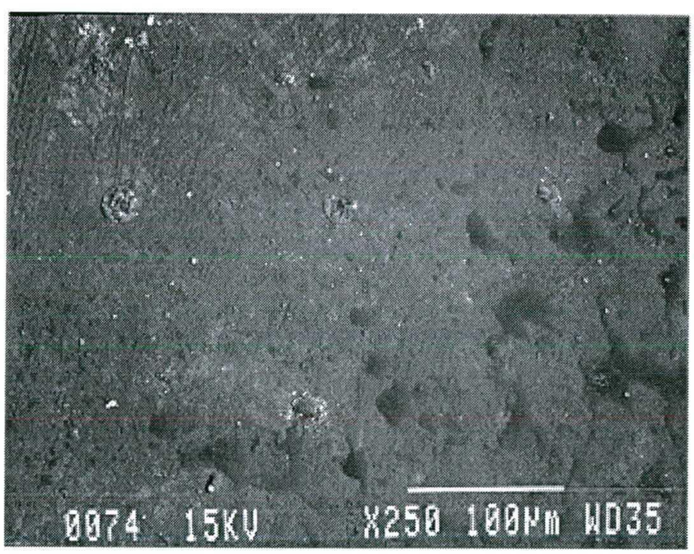


Figure 12. SEM photograph of the surface of a Contour bracket (*as received*).

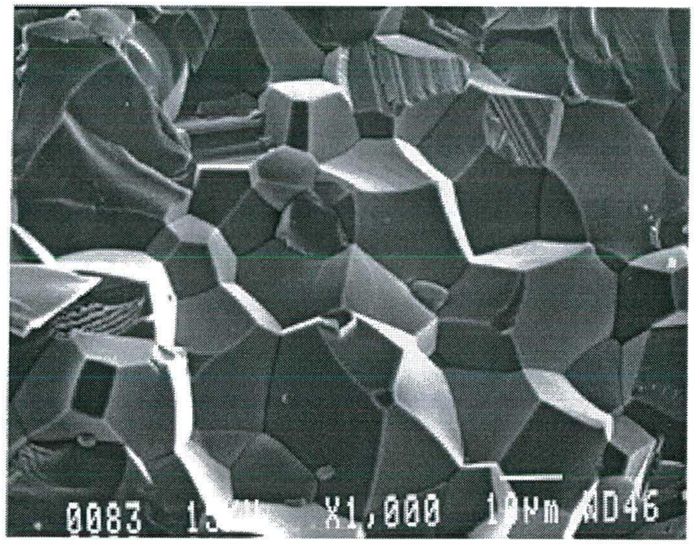


Figure 13. SEM photograph of the fracture surface of an Intrigue bracket.

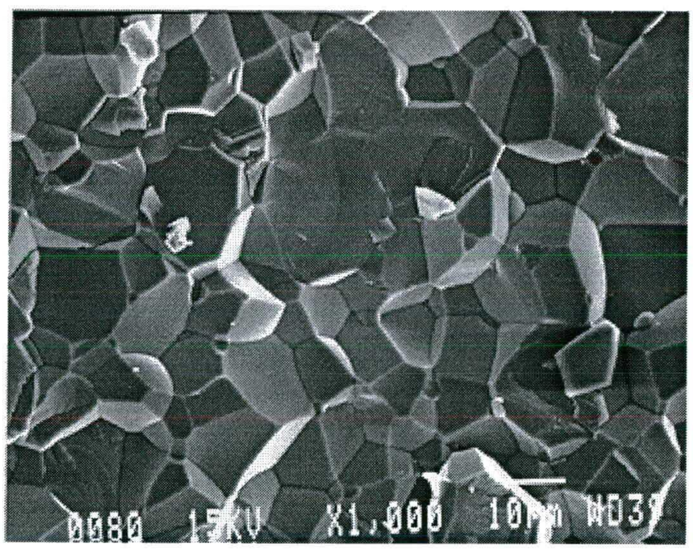


Figure 14. SEM photograph of the fracture surface of a Clarity bracket.

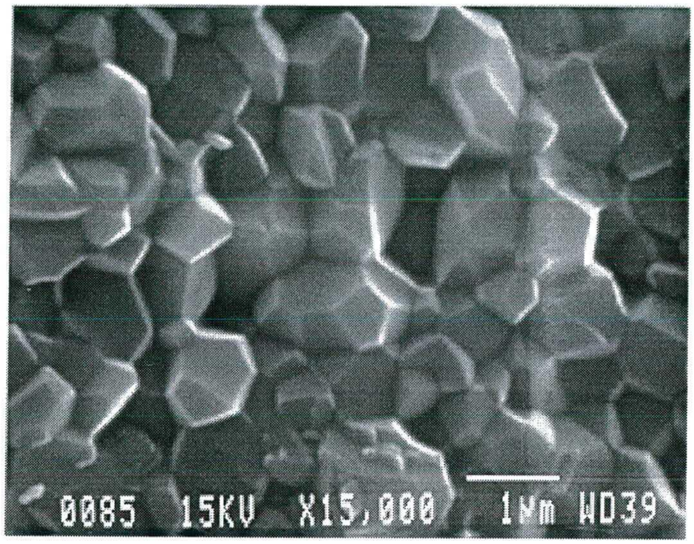


Figure 15. SEM photograph of the fracture surface of a Contour bracket.

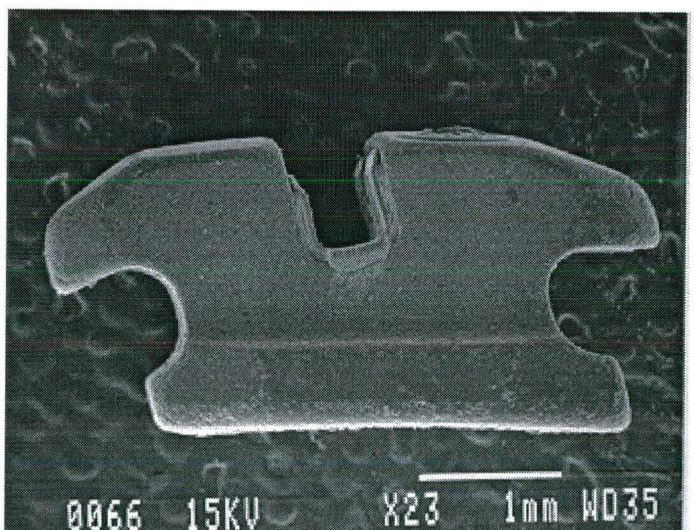


Figure 16. SEM photograph of a Clarity Bracket.

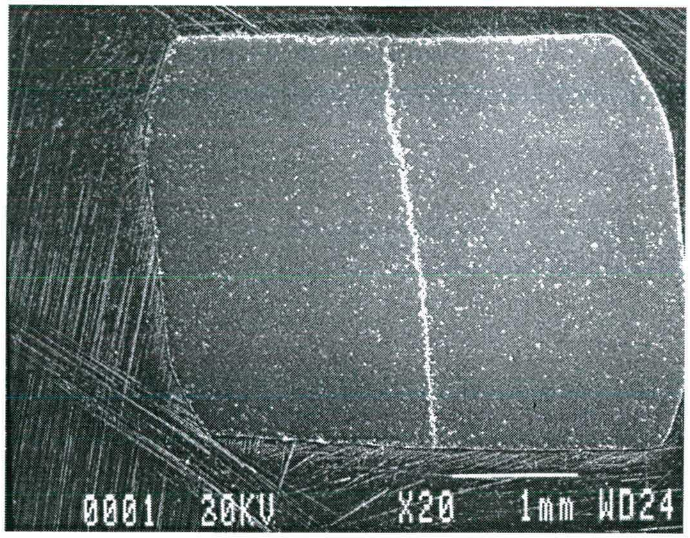


Figure 17. SEM photograph of the stress-concentrating defect in the base of the Clarity bracket (low magnification).

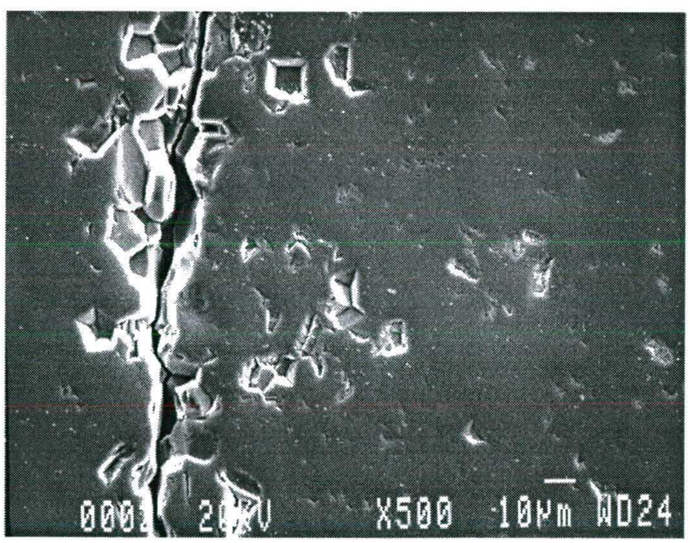


Figure 18. SEM photograph of the stress-concentrating defect in the base of the Clarity bracket (high magnification).

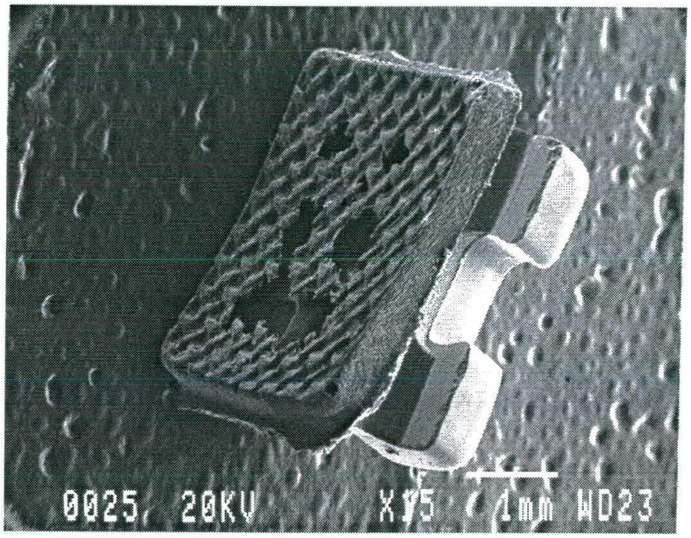


Figure 19. SEM photograph of the cast epoxy base of the MXi bracket.

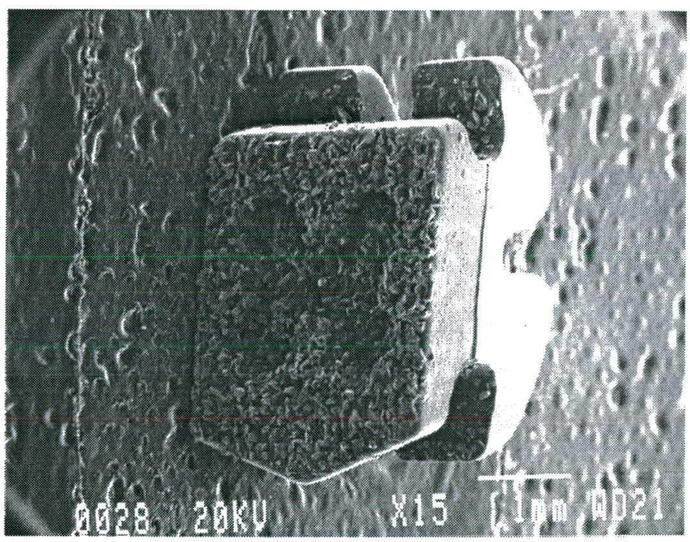


Figure 20. The Contour bracket base employs protruding crystals for mechanical retention.

Figure 21. The Intrigue bracket uses a groove-recessed retention mechanism.

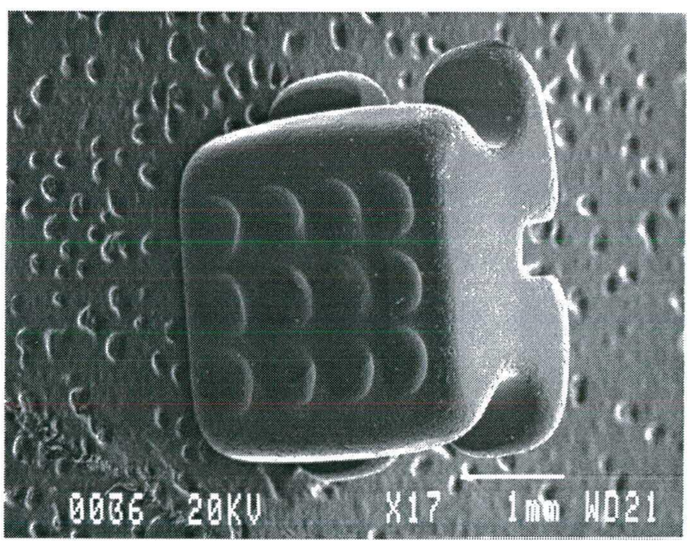


Figure 22. The Allure NSB bracket uses a dimple-recessed retention mechanism.

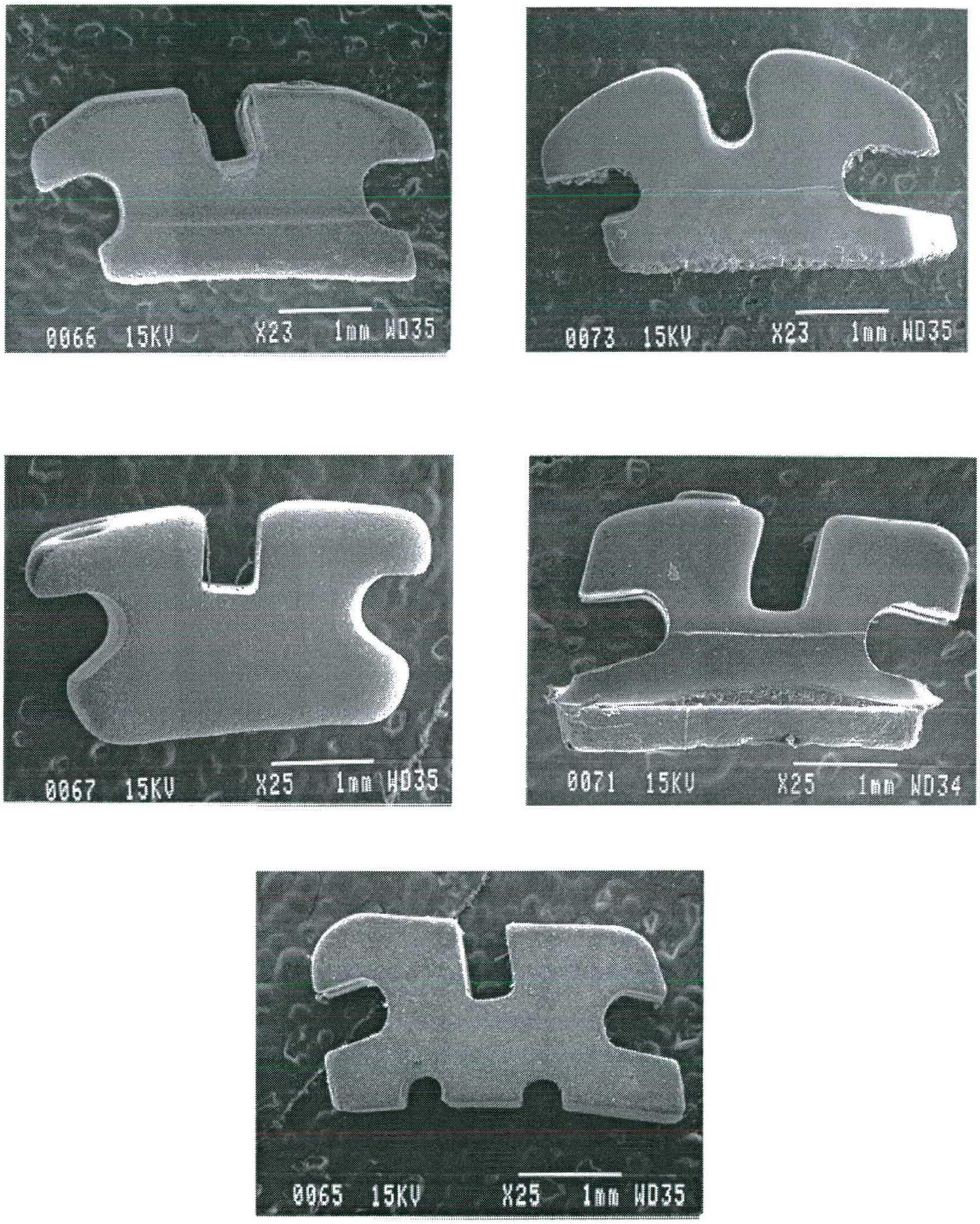


Figure 23. SEM photographs of *as received brackets* (profile view): A Clarity bracket (upper left); a Contour bracket (upper right); an Allure NSB bracket (left center); an MXi bracket (right center); an Intrigue bracket (bottom).

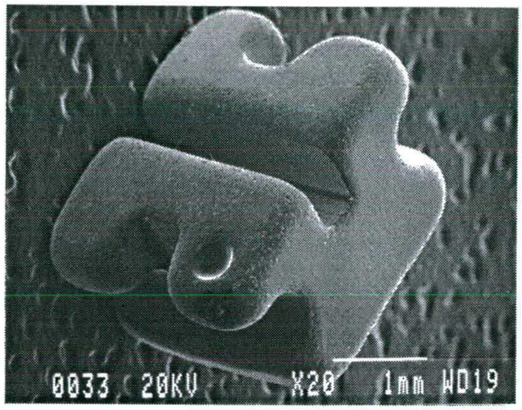


Figure 24. SEM photograph of an Allure NSB bracket: Archwire slot edge.

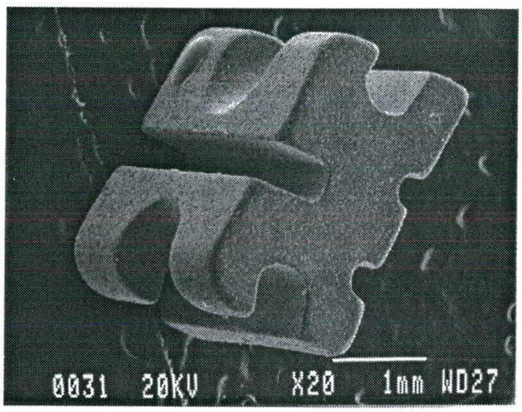


Figure 25. SEM photograph of an Allure NSB bracket: Archwire slot edge.

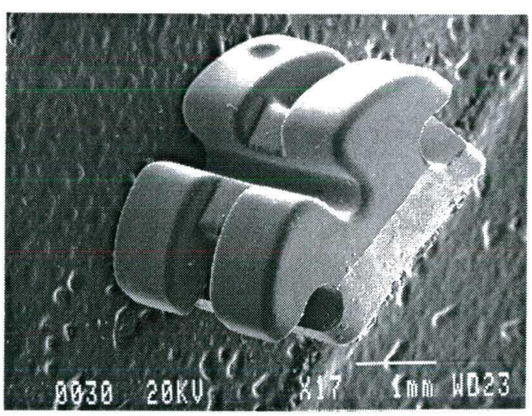


Figure 26. SEM photograph of a Contour bracket: Archwire slot edge.

APPENDIX C

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