

# Flexural strength of the joint between glass-infiltrated alumina frames and the alumina-magnesia modifier

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## Abstract

**Purpose:** The purpose of the present study was to evaluate the flexural strength of the joint between glass-infiltrated alumina frames and the experimental adjusting agent (MA modifier) that contains alumina and magnesia.

**Methods:** A commercially available adjusting agent (Optimizer), a slurry of alumina powder (Alumina modifier), and a bulk specimen (joint-free alumina) were used as controls. Beam-shaped alumina specimens were machined from an alumina block. The ends of two alumina beams were positioned at an interval of 1.0 mm and joined with each adjusting agent. The joined specimens were subjected to sintering, glass infiltration firing, glass control firing, and then a three-point bending test was carried out to evaluate the flexural strength.

**Results:** The maximum flexural strength was observed in the joint-free alumina, followed by MA modifier, Optimizer and Alumina modifier. With the exception for joint-free alumina, the failure modes after three-point bending test tended to shift from adhesive failure at substrate material-adjusting agent interface to cohesive failure within adjusting agent as the flexural strength increased.

**Conclusions:** The use of MA modifier significantly improved the flexural strength of joined glass-infiltrated alumina frame. The MA modifier could be applied for adjusting the margin as an alternative to Optimizer when fabricating crown and bridge substructures with In-Ceram Alumina system.

**Key words:** all-ceramic restoration, alumina, magnesia, CAD/CAM

## Introduction

All-ceramic restorations, such as crowns and fixed partial dentures, are useful to satisfy patients who desire esthetic dental treatment. In-Ceram Alumina (Vita Zahnfabrik, Bad Säckingen, Germany)<sup>1,2</sup> is a popular material used to fabricate coping structures for all-ceramic crowns using CAD/CAM systems. The liner shrinkage of In-Ceram Alumina is reported to be 0.21% after sintering,<sup>3</sup> and marginal adaptation is often necessary after machining. The marginal gap could be modified with a conventional adjusting agent (In-Ceram Alumina Optimizer, Vita Zahnfabrik; Optimizer). Optimizer is used to fill up small defects in crown and bridge substructures made with In-Ceram Alumina (Fig. 1). However, there is no information available regarding the bonding strength between the Optimizer and In-Ceram Alumina. In order to prevent all-ceramic restorations from marginal tipping, a strong bonding between the adjusting agent and the In-Ceram Alumina is required.

The fracture strength of joined In-Ceram Alumina is lower than that of joint-free In-Ceram Alumina.<sup>2</sup> The addition of magnesia controls the mechanical properties of glass-infiltrated alumina.<sup>4,5</sup> The authors have

previously reported that the addition of a trace amount of magnesia to In-Ceram Alumina slips (Vita Zahnfabrik) significantly improved the bonding to In-Ceram Alumina.<sup>6</sup> The purpose of the present study was to investigate the flexural strength of In-Ceram Alumina joined with an experimental adjusting agent containing alumina and magnesia.

### **Materials and methods**

The materials used in the present study are listed in Table 1. A total of 36 beam-shaped specimens (1.2 mm x 4 mm x 10 mm) were machined from In-Ceram Alumina GN-I, using a low-speed cutting saw (Isomet, Buehler Corp., Lake Bluff, IL, USA), and then ground with No. 600 silicon carbide paper.

The ends of the specimens were positioned at an interval of 1.0 mm. The specimen surfaces were moistened with distilled water, and then the gap was jointed with the adjusting agent using a brush. The jointed specimens were stored at room temperature for one hour after preparation and then sintered in a porcelain furnace (Austromat 3001, Dekema Dental-Keramiköfen GmbH,

Freilassing, Germany) (Table 2). In-Ceram Alumina Glass Powder (Vita Zahnfabrik) was first applied to the sintered specimens; the specimens were then placed on a platinum metal foil (Ishifuku Metal Industry Co. Ltd., Tokyo, Japan); finally, glass infiltration firing and glass control firing were carried out according to the firing schedule recommended by the manufacturer (Fig 2). Six beam-shaped specimens (1.2 mm x 4 mm x 20 mm) of In-Ceram Alumina GN-I were also prepared as controls (joint-free alumina).

In accordance with the standard (ISO 6872),<sup>7</sup> the bonded specimens were subjected to a three-point bending test on a universal testing machine (AGS-10kNG, Shimadzu Corp., Kyoto, Japan) at a cross-head speed of 0.5 mm/min. The test span was set at 15 mm and the jointed center of the specimens was brought to the cross-head point. Failure load was recorded in Newtons (N), and flexural strength (MPa) was calculated as follows:

$$M=3Wl/2bd^2$$

where  $W$  is the failure load (N),  $l$  is the test span (mm),  $b$  is the width of specimen (mm), and  $d$  is the thickness of specimen (mm).

The means and standard deviations of the flexural strength were

determined based on the results obtained for six specimens per group. The data were analyzed by one-way analysis of variance (ANOVA) and a Tukey compromise test at  $\alpha < .05$ .

After the three-point bending test, the debonded surfaces of all specimens were observed with an optical microscope (SMZ-10; Nikon, Tokyo, Japan) with a magnification of 20 $\times$ . Failure modes were categorized as: adhesive failure at substrate material-adjusting agent interface (A), cohesive failure within adjusting agent (C), complex adhesive failure at substrate material-adjusting agent interface and cohesive failure within adjusting agent (AC), and cohesive failure within substrate material (F).

## **Results**

The results of one-way ANOVA revealed the significant differences among the four groups ( $p < 0.05$ ). Table 3 shows the means and standard deviations of the three-point bending test. Tukey compromise test indicated that the mean flexural strengths of all groups were significantly different from each other.

The Alumina modifier showed the lowest flexural strength. The maximum and minimum flexural strengths were observed with the joint-free alumina and Alumina modifier, respectively. The MA modifier exhibited significantly higher flexural strength than the Optimizer. Except for one specimen that showed AC mode, all of the observed failure modes of MA modifier were C mode. In contrast, all specimens of Alumina modifier failed in completely in A mode. Half specimens joined with Optimizer failed in A mode and remaining half specimens failed in AC mode.

### **Discussion**

The present study revealed that In-Ceram Alumina frame was joined strongly with an experimental modifier containing alumina and magnesia, which indicates the advancement of dental material. The In-Ceram Alumina consists of two interpenetrating phases, alumina and lanthanum glass. When sintering, the alumina particles are partially fused together with contiguous particles to form porous alumina network, and lanthanum glass is infiltrated into the porous



alumina structure. This mechanism contributes to strong flexural strength of joint-free alumina in the present experiment. Therefore, control of interparticle fusion at the joined interface and infiltration of lanthanum glass into the adjusting agent could be a key factor in improving the flexural strength of the joined specimens.

With regard to MA modifier, it is considered that magnesia played an essential role. The concentration of magnesia 0.3% derived from the previous study.<sup>6</sup> It is reported that the addition of magnesia to alumina ceramics controls the distribution of alumina with a uniform particle size, crack bowing, crack deflection, and the wetting properties of the particle surface.<sup>4,5</sup> Growth of interparticle contacts improved flexural strength and fracture toughness of partially sintered porous alumina.<sup>8</sup> We therefore, speculate therefore that fusion of alumina particles to the In-Ceram Alumina surface was promoted by magnesia. It should be the reason for the difference observed between MA modifier and Alumina modifier. Distilled water was used instead of In-Ceram Alumina Mixing Liquid (Vita Zahnfabrik) as a component of MA modifier, because the shelf life of In-Ceram Alumina Mixing Liquid is relatively short.

The addition of In-Ceram Alumina Additive made handling easy in spite of no wax.

The failure modes observed in groups Optimizer, Alumina modifier, and MA modifier suggested that it tended to shift from adhesive failure at substrate material-adjusting agent interface to cohesive failure within adjusting agent as the flexural strength increased. When taking the flexural strengths for the partial cohesive failure within Optimizer into account, it is considered that cohesive strength of Optimizer was lower than that of MA modifier. The wax contents may affect the cohesive strength of Optimizer.

An ideal adjusting agent is desired to have some characteristics, such as high mechanical strength, easy handling, tooth color, strong bonding with In-Ceram Alumina and with feldspathic porcelain, durability against the thermal stress in oral environment. The present study showed that the flexural strength of In-Ceram Alumina joined with MA modifier is superior to that joined with Optimizer. Therefore, further evaluation should be pursued for clinical application.

## **Conclusions**

Within the limits of the 24 h three-point bending test, it is concluded that In-Ceram Alumina joined with MA modifier has a significantly higher flexural strength than joined with Optimizer or Alumina modifier. MA modifier could potentially be applied for fabricating all-ceramic restorations in conjunction with glass-infiltrated alumina.

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Table 1 Materials used in the present study

Name	Component	Manufacturer	Lot Number
<b>Substrate material</b>			
In-Ceram Alumina GN-I		Vita Zahnfabrik, Bad Säckingen, Germany	7743
<b>Adjusting agent</b>			
Optimizer	Alumina, Wax	Vita Zahnfabrik	7481
MA modifier	Liquid/Powder ratio = 0.27		
	<u>Powder</u> : In-Ceram Alumina Powder 99.7%	Vita Zahnfabrik	26270
	Magnesia 0.3%	Wako Pure Chemical Industries Ltd., Osaka, Japan	KLH1020
	<u>Liquid</u> : In-Ceram Alumina Additive 1.0%	Vita Zahnfabrik	7608
	Distilled water 99.0%		
Alumina modifier	In-Ceram Alumina Powder 78.7%	Vita Zahnfabrik	26270
	Distilled water 21.3%		
<b>Infiltration glass</b>			
In-Ceram Alumina Glass Powder		Vita Zahnfabrik	7745

Table 2 Schedules for sintering, glass infiltration firing, and glass control firing

	Drying	Starting temperature	Heating rate	Ultimate temperature	Holding
	(min)	(°C)	(°C/min)	(°C)	(min)
Sintering 1*	20	200	76	1140	40
Sintering 2**	20	200	76	1140	120
Glass infiltration firing***	8	600	40	1110	120
Glass control firing***	2	600	80	960	10

\* Sintering schedule for the Optimizer, Alumina modifier and joint-free alumina.

\*\* Sintering schedule for MA modifier.

\*\*\*Firing schedules for all specimens.

Table 3 Flexural strengths and failure modes after the three-point bending test

Adjusting agent	Flexural strength (MPa)		Failure mode**
	Mean*	SD	(Number of specimens)
Optimizer	263.2	42.3	A(3) AC(3)
Alumina modifier	156.1	54.0	A(6)
MA modifier	415.7	69.3	AC(1) C(5)
Joint-free alumina	510.1	29.5	F(6)

\*All groups were significantly different from each other ( $\alpha < .05$ ).

\*\*A: Adhesive failure at substrate material-adjusting agent interface; C: Cohesive failure within adjusting agent; AC: Complex adhesive failure at substrate material-adjusting agent interface and cohesive failure within adjusting agent, F: Cohesive failure within substrate material.

### **Figure legends**

Fig. 1 The coping machined from In-Ceram Alumina GN-I using a CAD/CAM system (Left). Adjustment of the marginal gap was completed with In-Ceram Alumina Optimizer (Right).

Fig. 2 A pair of beam-shaped specimens machined from In-Ceram Alumina GN-I using a CAD/CAM system (lower). A specimen consisting of beams joined with MA modifier prepared for the three-point bending test. Sintering, glass infiltration firing, and glass control firing were completed (upper).



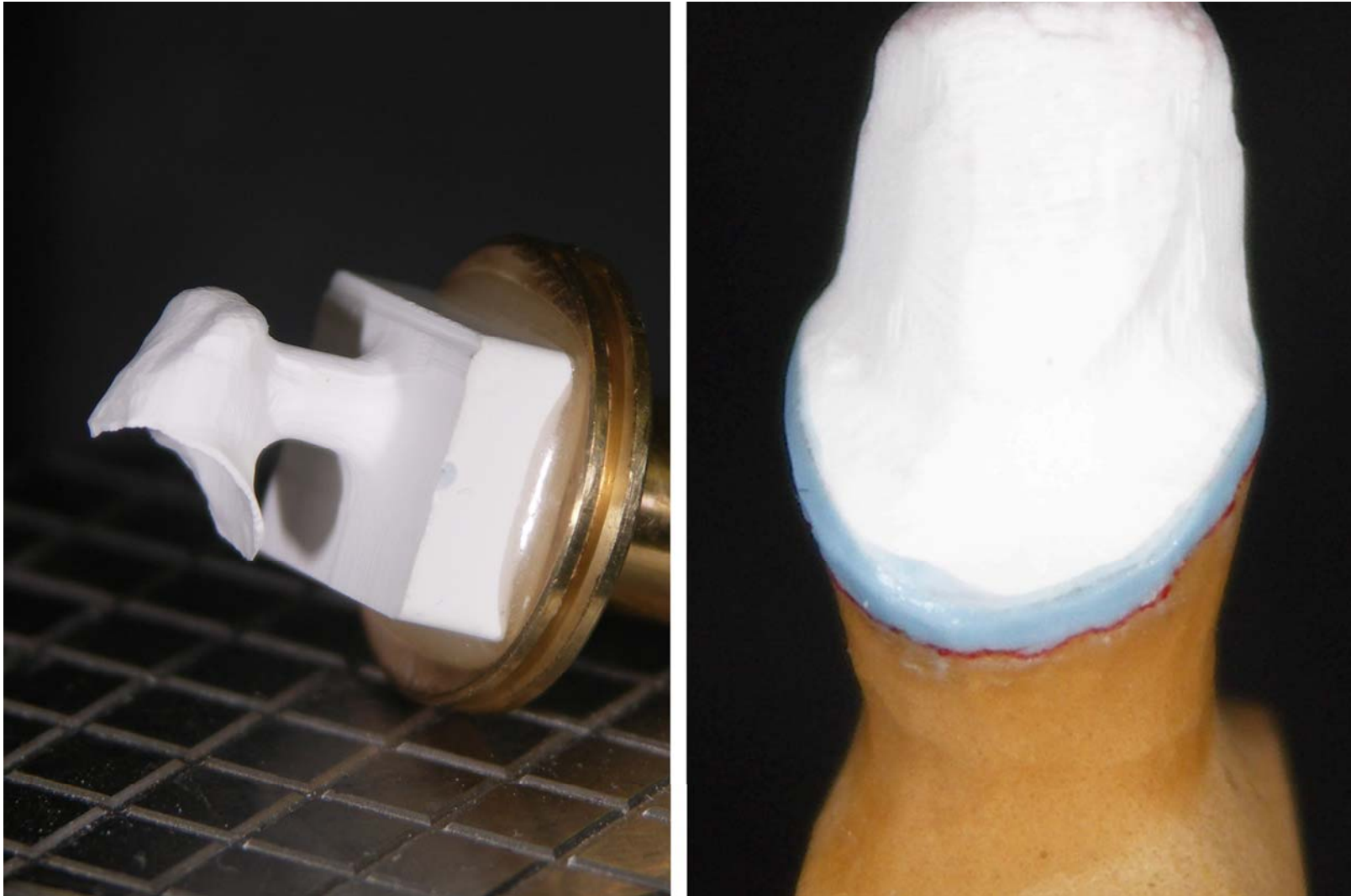


Fig. 1

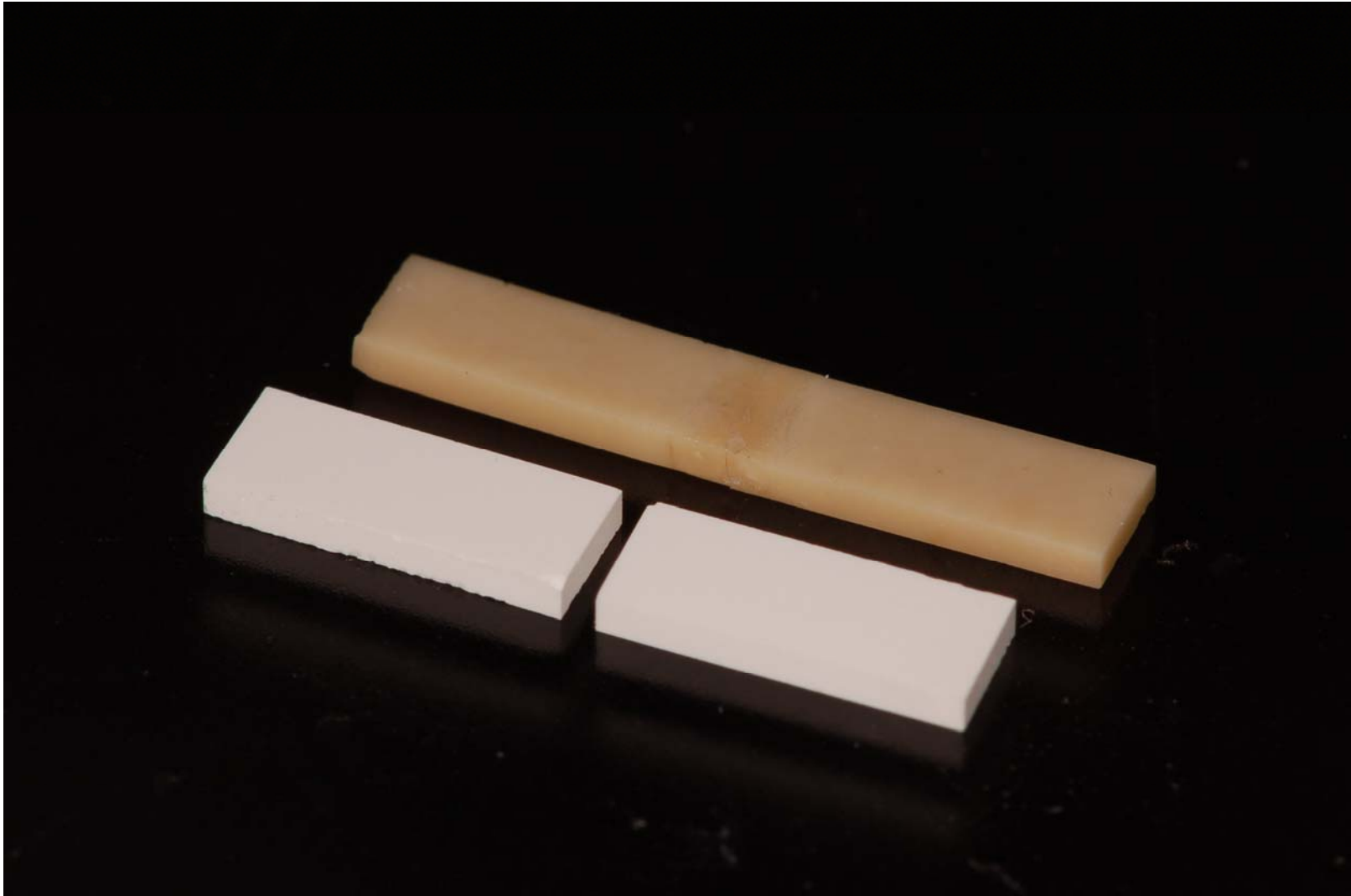


Fig. 2