# NATIONAL BUREAU OF STANDARDS REPORT

## NBS PROJECT

311.05 - 11 - 3110561

## June 30, 1970

## NBS REPORT

10 274

**Progress Report** 

#### on

## RADIOPAQUE DENTURE BASE MATERIALS **TECHNIC DENTURES**

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This investigation was supported in part by Research Grant DE02742-01 to the American Dental Association from the National Institute of Dental Research and is part of the dental research program conducted by the National Bureau of Standards in cooperation with the Council on Dental Research of the American Dental Association; the Dental Research Division of the U. S. Army Medical Research and Development Command; the Dental Sciences Division of the School of Aerospace Medicine, USAF the National Institute of Dental Research; and the Veterans Administration.

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U.S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

#### RADIOPAQUE DENTURE BASE MATERIALS

#### TECHNIC DENTURES

#### ABSTRACT

Technic dentures were made using composite base materials containing 30, 40 and 50 weight percent of a silane-treated, radiopaque, powdered glass as the reinforcing filler and poly(methyl methacrylate) as the matrix. Dentures containing no glass were included for comparison. Molar-to-molar dimensional changes, effects of silane treatment of the porcelain teeth and handling and finishing characteristics were determined.

Dentures containing glass had less dimensional change during curing and subsequent water storage than those without glass. Thick upper dentures exhibited the least change and thin lowers, the most. The differences in the magnitudes of the changes would not be clinically noticeable.

Silane treatment of the porcelain teeth in thick upper dentures resulted in breakage of the posterior teeth in almost all instances. Posterior teeth also fractured in thick upper dentures containing 40 and 50% of the silanetreated glass even though the teeth were not treated with silane. Mixing and molding properties of the composite materials, while acceptable, were slightly inferior to the material containing no glass. Finishing characteristics were definitely inferior in that it was more difficult and timeconsuming to attain the final surfaces and these surfaces were not as smooth as those in dentures without glass.

## RADIOPAQUE DENTURE BASE MATERIALS:

#### TECHNIC DENTURES

Introduction

A previous report described the need for esthetically acceptable radiopaque denture base materials.<sup>1</sup> It was established in a review of 123 cases of ingested and aspirated dentures and fragments that radiographic localization of the foreign bodies often would have been facilitated if the base material had been radiopaque.

Another report described the development of an experimental denture base material based on the addition of a radiopaque, silane-treated glass powder to poly(methyl methacrylate) (PMMA).<sup>2</sup> Other resin-glass combinations were investigated but the above material appeared to offer the best combination of properties for immediate development. Specimens containing from 29 to 57 weight percent of the glass filler had sufficient radiopacity to be of aid in localization of denture foreign bodies. Subsequently, pertinent physical properties of denture base materials composed essentially of PMMA and 0, 30, 40, and 50 weight percent of the radiopaque glass powder were reported.<sup>3</sup>

In the present study twenty technic dentures were made of the foregoing materials to determine the molar-to-molar dimensional change during processing and water storage; the effects of silane treatment of porcelain teeth when used with composite materials of these types, and the mixing, molding, and finishing characteristics during denture fabrication.

Materials and methods\*

The following materials were used in making the dentures: 1. Liquid - The monomer liquid was 98.5% methyl methacrylate (Rohm and Haas Co.) and 1.5% ethylene dimethacrylate (Borden, Inc) by weight.

\* Certain commercial materials and equipment are identified in this paper to specify adequately the experimental procedure. In no instance does such identification imply recommendation or endorsement by the National Bureau of Standards or that the material or equipment identified is necessarily the best available for the purpose.

- 2. <u>Powder</u> The powder was 95.0% PMMA and 5.0% dibutyl phthalate by weight (Esschem Co., Div. of Sartomer Resins, Inc. Type #P5-69-203).
- 3. <u>Glass fillers:</u> <u>Filler A</u> A silane-treated powdered glass (Corning Glass Works, Code X816JL) that was elutriated before use in an attempt to remove the smallest particles. It had a batch formulation as follows:  $SiO_2$ , 44;  $BaF_2$ , 28;  $B_2O_3$ , 16; and  $Al_2O_3$ , 12, in mole percent.<sup>4</sup> The powder particle sizes ranged from about 1 to 50  $\mu$ m. The silane treatment and elutriation procedure have been described previously.<sup>2</sup>

Filler B - Two dentures were made using Filler B after it had been washed in an attempt to remove any excess silane. The washing was carried out by making a thin slurry of the powder in 95% ethanol. The slurry was vigorously shaken for several minutes and then vacuum filtered. This process was repeated using absolute ethanol, and finally acetone. The powder was dried and passed through a U. S. Standard Sieve No. 100 to remove any lumps of caked or agglomerated material.

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<u>Filler C</u> - One denture was made with the glass as received without silane treatment or elutriation.

- 4. <u>Pigment and opacifier</u> Most of the dentures were pigmented with cadmium red (United Color and Pigment Co.) and opacified with titanium oxide (Fisher Scientific Co.).
- 5. <u>Silane</u> (Union Carbide Al74 Silane) Fillers A and B and some of the porcelain denture teeth were treated with a silane coupling agent to promote bonding to the resin of the base material. One denture was made with the monomer liquid to which was added 3% by weight of the unhydrolyzed silane.
- 6. <u>Porcelain teeth</u> Technic teeth (Trubyte Bioform mold 42-F with Pilkington Turner Posteriors, Dentsply International, Inc.) were used in the dentures made for the purpose of measuring molar-to-molar dimensional change. Regular clinical teeth of the same mold were used in the dentures made to study the effects of silane treatment of the teeth.

The dentures tested contained either 0, 30, 40, or 50 weight percent of the radiopaque glass. The composition of the mix for each glass content was reported previously.<sup>3</sup>

Twelve complete dentures with either no filler or Filler A were made to determine the molar-to-molar dimensional change occurring during processing and subsequent water storage. The dentures were of three different types; a thick upper, a thick lower, and a thin lower for each of the four glass levels; the volume of the base material in each denture type was approximately 20, 25, and 11 ml, respectively.

The gypsum casts and wax models for each denture type were made in the same rubber molds to ensure nearly exact duplications. Dental stone was used for pouring the casts and for all flasking procedures. Wax was removed by the customary boil-out techniques after which the teeth and flasks were thoroughly cleansed with boiling water followed with chloroform. All dentures were cured at 73°C for 9 hours and allowed to cool in the curing bath to room temperature.

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The method for determining the molar-to-molar dimensional change has been employed previously.<sup>5</sup> Basically, it involved measurement of the distance between reference marks on the ends of stainless steel pins that had been ' cemented in the occlusal surfaces of the second molar denture teeth. The measurements were made at 23 ± 1°C with a measuring microscope and recorded to the nearest 0.0025 mm at the following stages: waxed denture flasked to the occlusal surfaces; cured denture deflasked to the occlusal; denture removed from cast; denture finished and stored in water for one week, and denture stored in water for one month.

Eight additional thick upper dentures were made to determine the effect of silane treatment of clinical porcelain teeth in dentures made with fillers A, B, and C. The teeth that were silane-treated and the fillers are listed in Table 1.

The teeth were prepared for silane treatment by washing with a boiling solution of detergent in water followed by a thoroughrinse with boiling distilled water. A 0.5% solution of the silane in distilled water was acidified to pH 3-4 with

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glacial acetic acid and stirred for one hour. The cleaned teeth were placed in the solution for 30 minutes, drained, and baked at 110°C for 15 minutes. The above treatment was carried out before wax-up of the dentures. Flasking, packing, curing, and finishing were carried out in the usual manner except that extreme care was exercised to ensure that the teeth were completely cleaned during the boil-out procedure. Chloroform was not used.

After finishing, all twenty dentures were inspected for broken teeth. The dentures were then temperature cycled by placing them in 5°C water for 5 minutes followed by immersion in 55°C water for 5 minutes. This cycle was repeated 5 times after which the dentures were again inspected for broken teeth. The twelve dentures made for measuring molar-to-molar dimensional change were not temperature cycled until all measurements had been completed.

Results and discussion

Molar-to-molar dimensional change: The values for the molar-to-molar dimensional changes as determined at the various stages may be seen in Figure 1. Each point on the graph

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represents the average percent dimensional change for the thick upper, thick lower, and thin lower dentures made with each material. The magnitudes of the overall changes range from -0.32% for the 0% glass dentures to -0.24% for the 50% glass dentures. Such differences might be expected due to the lower coefficients of thermal expansion of the glass-containing materials.<sup>3</sup>

The greatest contraction occurred with the thin lower dentures and the least with the thick upper dentures. For example, the thin lower dentures containing 40% glass contracted 0.50% and the thick upper dentures containing 40% glass contracted 0.19%. Evidently, denture thickness and shape can act to restrict the contractural forces. The greatest contraction occurred when the dentures were removed from the casts. Prior to removal, the contraction was restricted by the cast and flasking stone. After finishing, the dentures continued to contract probably because of continuing stress relief, but soon began to expand due to water sorption.

The overall differences among the four materials was only slight and probably would have no clinical significance. A previous investigation<sup>5</sup> has shown that the molar-to-molar

linear dimension of heat-cured PMMA complete technic dentures will decrease about 0.45 to 0.55 percent during curing. Most of this change can be attributed to the thermal contraction that occurs when the dentures are cooled from curing temperature to room temperature. Part of the contraction is compensated for by the subsequent expansion that occurs due to water sorption and thermal expansion when the denture is inserted in the mouth. The overall dimensional change can be expected to be a slight shrinkage with the heat-cured PMMA type resins. Subsequent changes that do occur are not clinically noticeable because the adaptability and change in the supporting tissues is of much greater magnitude.<sup>5</sup> Nonetheless, it can be assumed that the more nearly the overall dimensional change approaches zero, the better the denture will duplicate the impression.

Silane treatment of porcelain teeth: The advantages of constructing dentures in which the porcelain teeth are bonded to the base materials are well recognized. One advantage of such bonding is the prevention of seepage of oral fluids into the crevice between the teeth and the base material.<sup>6</sup> These fluids cause the development of stains

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and odors which contribute to poor denture hygiene. Another advantage may be a strengthening of the denture<sup>6</sup> by effectively increasing its cross-sectional area. Bonding would eliminate the notches in the resin due to unbonded teeth, and thus some of the stress concentrations that occur in these areas from notch effects.

Such bonding is made possible by applying an appropriate silane coupling agent to the teeth before curing the denture.<sup>6-8</sup> However, the effects of the bonding are such that stresses are introduced into the teeth resulting in severe weakening or fracture. These stresses arise as a result of the differential in thermal expansion when bonding occurs between the resin base materials  $[70 - 116 \times 10^{-6}/^{\circ}C (37-70^{\circ}C)]^{9}$  and the porcelain teeth [8.0  $\times 10^{-6}/^{\circ}C (30-400^{\circ}C)]^{10}$ . Therefore, silane treatment has not become a clinically acceptable procedure for posterior teeth.

Because of the lower coefficients of thermal expansion of the composite bases used in this investigation [58 - 72 x  $10^{-6}/°C$  (27-70°C)],<sup>3</sup> it was hoped that porcelain teeth might be bonded to the base materials without weakening or fracturing of the teeth.

However, when the teeth on the right side of the dentures were silane treated, the posterior teeth on that side fractured in almost all cases (Table 1, dentures 1 - 4). The fractures of the posterior teeth occurred in a mesial-distal direction in the central grooves in all cases (Fig. 2). (No breakage of anterior teeth occurred in any of the dentures made in this investigation or in another investigation.<sup>6</sup>) In addition it was noted that in the 40 and 50% glass-containing dentures (Table 1, dentures 3 - 4) numerous teeth fractured that had not been silane treated. An examination of the twelve dentures made for measuring molar-to-molar dimensional change showed that seven out of eight of the posterior technic teeth in the 50% glass, thick upper denture were broken although the teeth had not been silane treated. This suggested that there might have been excess silane in the glass powder from its original silane treatment and that this was effective in bonding the teeth to the base, thereby causing the unexpected tooth breakage.

To determine if washing the glass before use would prevent tooth breakage, two dentures were made with Filler B (washed). One of these dentures was made without any of the

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teeth silane treated (Table 1, denture 5) and one with all the teeth silane treated (Table 1, denture 6). Most of the posterior teeth fractured, especially after temperature cycling, in both dentures. Perhaps the washing procedure was not effective in removing the excess silane and this may have allowed some to migrate to the surface of the porcelain teeth. It is also possible that the higher moduli<sup>3</sup> of the materials containing silane-treated glass contributed to the stresses applied to the teeth.

There were no fractured teeth in the denture made with glass that had not been silane treated (Filler C), Table 1, denture 7. Placement of 3% by weight of the silane in the monomer also did not result in fractured teeth (Table 1, denture 8).

Many of the dentures were sectioned by making a cut along the tissue surface of the posterior ridge area as deep as possible without touching the teeth. The base material buccal to the posterior teeth was then fractured away. When this was done in those dentures containing fractured teeth, many of the teeth were observed to have fractured into numerous pieces with some of the fragments adhering securely to the base material as in Figure 3. In sectioning the denture made with untreated glass the teeth could be removed easily with no apparent adhesion or tooth damage (Fig. 4).

Mixing, handling, and finishing properties: In making mixes containing the reinforcing glass filler, there was a tendency for the glass to settle to the bottom of the mixing vessel. In order to obtain an even distribution of the glass, it was necessary to stir the materials more often than would be done for a mix containing no glass, especially during the early stages when the mix was relatively fluid.

When the liquid was added to the powders, it appeared at first that the mix was too dry, but with continued stirring, it became fluid enough to ensure proper wetting of the polymer beads.

The molding characteristics of the glass containing materials were similar to those with no glass. However, they did not seem to flow quite as readily under similar flasking pressures. In addition, the glass-containing

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materials (especially 50% mixes) tended to adhere slightly to the flasking stone and polyethylene separating sheets. Alginate mold lining compounds worked well with these materials.

Finishing of the glass-containing dentures was more difficult and time-consuming, but could be accomplished using accepted techniques. Shaping and trimming the gingival areas was especially difficult and time-consuming because the material tended to adhere to the porcelain teeth and did not cut easily with hand finishing instruments. Finishing procedures using a wet rag wheel and pumice were also time-consuming because deep scratches rag were hard to remove. Final polishing with a dry/wheel and a polishing agent did not produce as glossy a finish as that obtained on the dentures without glass.

## Summary and conclusions

Twenty technic dentures were constructed of base materials containing 0, 30, 40, or 50 weight percent of a silane-treated, radiopaque glass powderas the reinforcing filler and poly(methyl methacrylate) as the resin matrix.

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The overall average molar-to-molar dimensional change of three dentures for each material ranged from -0.24 for the 50% glass-containing dentures to -0.32 for those containing no glass. The greatest change occurred in thin lower dentures with no glass and the least in thick upper dentures with 50% glass. The addition of glass reduced the overall dimensional change, but the amounts would not be clinically noticeable by the patient or the dentist.

Although the coefficient of linear thermal expansion of the glass-containing materials is significantly less than materials containing no glass,<sup>3</sup> it did not prevent the breakage of posterior porcelain teeth that had been silane bonded to the base materials. In addition, there was considerable breakage of posterior teeth in thick upper dentures containing the higher percentages of silanetreated glass even though the teeth were not intentionally silane treated. Washing the treated glass with ethanol and acetone before use was not successful in preventing tooth breakage. There were no broken teeth in the denture made with

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glass that had not been silane treated and there was no instance of fracture of any anterior teeth in any of the dentures.

It was demonstrated in a previous paper that the greatest need for radiopaque denture base materials was in maxillary partials consisting of a resin palate to which is attached one or more anterior teeth.<sup>1</sup> Since there usually is no labial flange in these partials, one might expect very low stresses to be applied to the teeth even when they are silane bonded to the base material. If posterior teeth with a buccal flange are present, then fracture or weakening of the teeth is probable. An alternative would be to use radiopaque acrylic resin teeth bonded to the composite denture base.

Mixing and molding characteristics of the glasscontaining materials, while acceptable, were slightly inferior to the material containing no glass. Finishing characteristics were definitely inferior in that it was more difficult and time-consuming to attain the final surface and this surface was not as smooth as in dentures containing no glass.

#### TABLE 1

#### FRACTURING OF PORCELAIN TEETH IN DENTURES

Denture No.*	Teeth Silane Treated	Filler Used	Fractured Teeth Before Cycling†	Fractured Teeth After Cycling*
l	2-8‡	None	2-5‡	2-5‡
2	2-8	30% Filler A	2,4	2-4
3	2-8	40% Filler A	2-5, 13, 14	2-5, 13, 14
4	2-8	50% Filler A	2-5, 13-15	2-5, 13-15
5	None	50% Filler B	4, 12, 14	2-5, 12-14
6	All	50% iller B	2, 3, 4, 14	2-5, 13-15
7	None	50% Filler C	None	None
8	None 3% Silane in monomer	None	None	None

\* All dentures were thick uppers

- <sup>†</sup> Cycling consisted of immersing the entire denture in 5°C water for five minutes followed by immediate insertion in 55°C water for five minutes. The cycle was repeated five times.
- \* Tooth numbers refer to those in the system adopted by the Council on Dental Education of the American Dental Association whereby tooth number 1<sup>k</sup> the upper right 3rd molar, tooth number 16 is the upper left 3rd molar, tooth number 17 is the lower left 3rd molar, and tooth number 32 is the lower right 3rd molar. Porcelain teeth corresponding to numbers 1 and 16 were absent.

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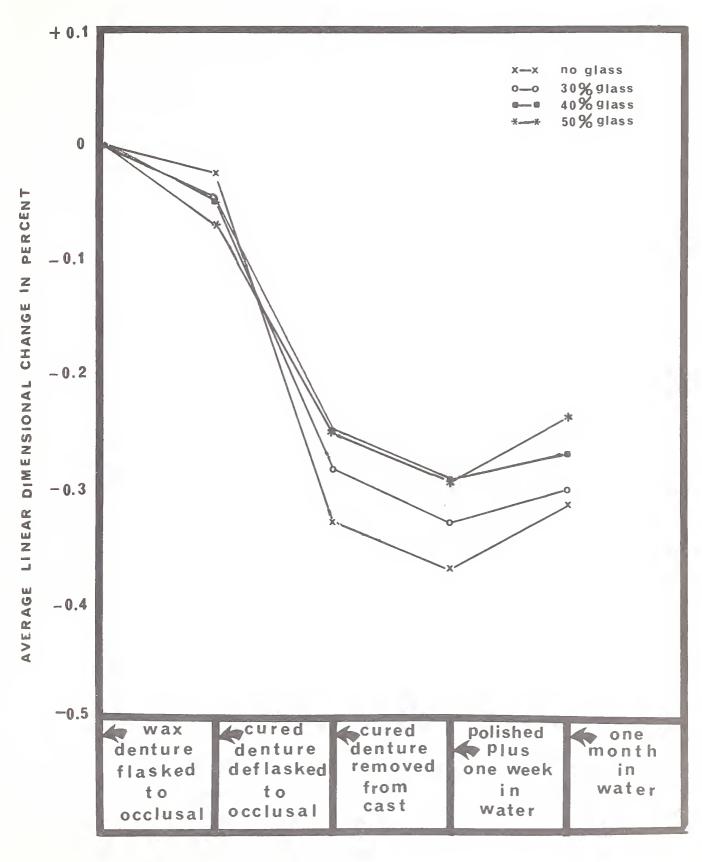
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Legends

- Fig 1: Average molar-to-molar dimensional change of technic dentures. Each point represents the average percent change for a thick upper, a thick lower, and a thin lower denture for each material.
- Fig 2: A section cut from a 40% glass complete upper denture showing mesiodistal fractures in the central groove of the posterior teeth. The teeth were silane treated.
- Fig 3: Section removed from a 40% glass denture showing the numerous fractures that occurred upon removal of the bulk of the teeth leaving some portions securely attached to the base material.
- Fig 4: Section removed from a 50% glass denture in which the glass had not been silane treated. The teeth show no evidence of bonding to the base material.

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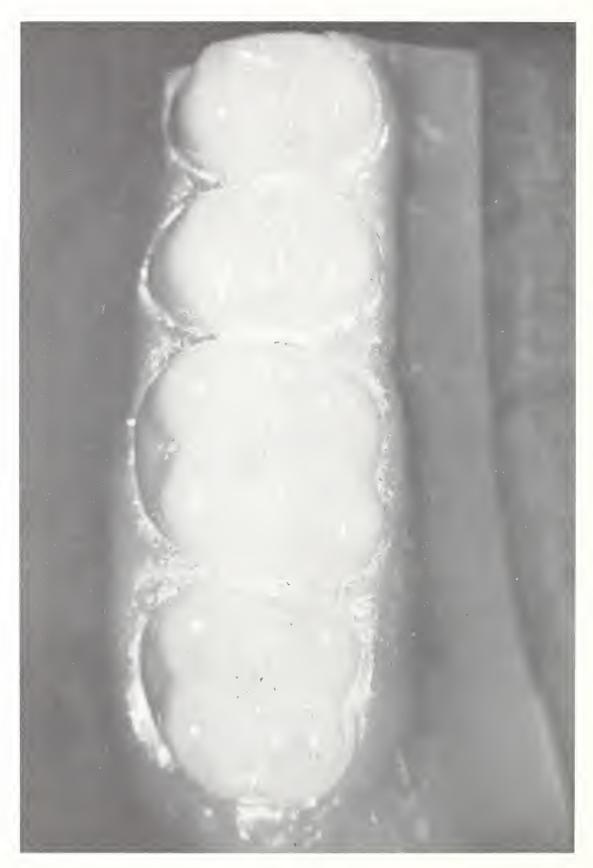
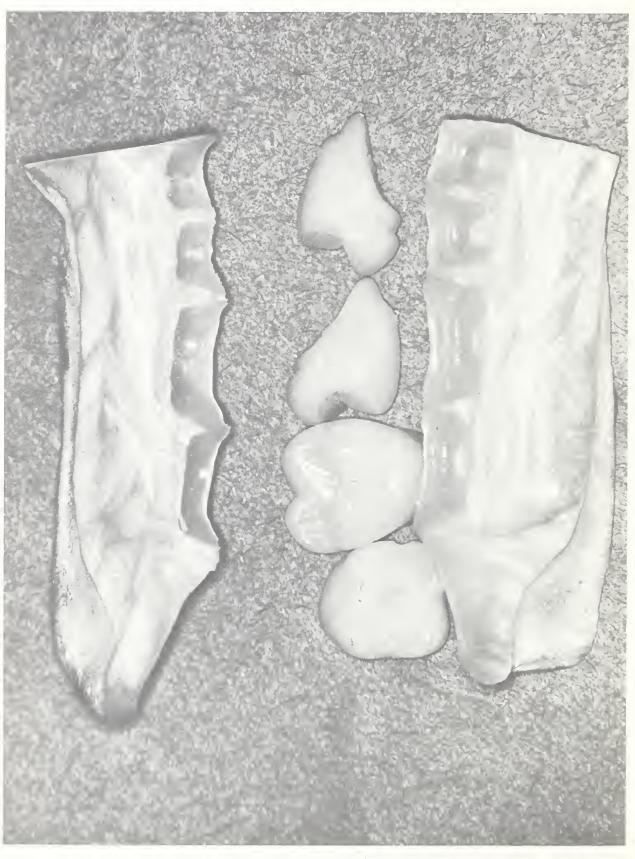


fig 2



fig 3



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