

solutions of various weights of poly(dimethylsiloxane), trimethylsiloxy terminated, 1000 CS, in cyclohexane, and shaped into discs by pressing in a 12.6 mm die at 428 megapascals for 30 seconds.

The discs were fired for 10 minutes at temperatures ranging from 550°–745° C. The resulting discs were porous and were silanated with 3-methacryloxypropyltrimethoxysilane and then infiltrated with methyl methacrylate activated with a chemical initiator system (2 wt % benzoyl peroxide and 0.5 wt % N,N-dihydroethyl-p-toluidine).

After 24 hours ambient (22° C.) polymerization, the excess polymer was removed and the resulting easily machinable discs were tensile tested in biaxial flexure. Initially a 2x3 factorial design with n=3–8 per group was used to assess the effects of the two factors, temperature (for 10 minutes) at two levels: low temperature (655° C.) and high temperature (715° C.) and binder concentration at three levels: 0 wt %, 5 wt % and 20 wt %. Temperature and binder concentration were both significant (p<0.001). The interaction between temperature and concentration was not significant (p>0.76). Duncan's Multiple Comparison (alpha=0.1) test was used to determine specific differences among the three concentrations. Significant differences were found between 0 and 5 wt % and between 5 and 20 wt %.

Example 2

Based on the results of Example 1, the 5 wt % binder was studied at temperatures of 550° C., 600° C., 655° C., 685° C., 715° C. and 745° C. As a result, it was found that optimum temperature for the 5 wt % binder appears to be about 655° C. yielding an IPC with a mean tensile strength of 140 MPa. The ceramic powder used in this example was a leucite containing feldspathic dental porcelain used for metal-ceramic dental restorations (Ceramics II, incisal light, Johnson & Johnson, Inc.). The poly(dimethylsiloxane) was trimethylsiloxy terminated and had a viscosity of 0.001 m²/sec (Petrauch Systems, Bristol, Pa.). Cyclohexane was used as a solvent, or carrier, for the siloxane.

Individual test specimens were prepared from 0.5 grams of feldspathic porcelain and 14 drops (approx. 0.7 ml) of the siloxane solution. Concentrations of pre-ceramic polymer were evaluated for 0%, 5% or 20% by weight. Powder specimens were pressed as in Example 1. Pressed powder discs were then fired in air for 10 minutes at one of six temperatures mentioned above ranging from 550° C. to 745° C.

The porous, pressed discs were silane treated with a common coupling agent. 3-methacryloxy-propyltrimethoxy silane. Silanation was used both to prepare the interparticle-necked ceramic mixture for monomer infiltration as well as to maximize later physical properties.

Following silanation, specimens were infiltrated as in Example 1. A 24 hour ambient temperature cure was followed by a 65° C. post-cure or annealing.

Excess polymer was removed from the discs using rotary instruments and abrasive paper. Specimens were finished only through 600 grit, and were therefore not highly polished. Discs were tested using biaxial flexure in a pin on 3 ball fixture at a crosshead speed of 0.5 mm/min. Failure stresses were calculated using the equation of Wachman (*J. Mater. Sc.*, 7:188–194, 1972; herein incorporated by reference).

As can be seen from Table 1 below, the experimental matrix consisted of 72 specimens distributed over three different pre-ceramic polymer concentrations and six

temperatures, ranging from 550° C. to 745° C. It can be seen that the specimens were not uniformly distributed over the matrix.

TABLE 1

Pyrolysis Temperature	0 wt %	5 wt %	20 wt %
550	2	—	2
600	4	7	—
655	7	9	3
685	4	8	—
715	3	8	3
745	4	8	—

Experimental matrix - number of specimens per pyrolysis temperature and weight percent of poly(dimethylsiloxane) polymer in cyclohexane.

Results

FIG. 1 shows mean fracture stresses for each polymer concentrations as a function of pyrolysis temperature. ANOVA and a 95% multiple range test were used to evaluate significant differences among conditions. Within the 5 weight percent group, the 655° C. specimens differed from all others except those pyrolyzed at 600° C. In addition there were significant differences between both the 685° and 715° C. specimens compared with the 745° C. specimen group. Within the 0 weight percent group, only the two lowest temperature sets differ from the two highest temperature sets. Within the 20 weight percent group there was no statistical difference, probably in part due to the rather small number of specimens. Between concentration groups at 655° C., the 5 weight percent set differs from the 0 and 20 weight percent sets. Overall, the low temperature specimen groups were significantly stronger than the high temperature sets and a maximum may exist when the pre-ceramic polymer was used at an intermediate temperature.

FIG. 2 is a three-dimensional response surface fit to the existing data set, having a degree of freedom adjusted R² equal to 0.85. Fracture stress, as the response variable, was plotted on the z axis versus pyrolysis temperature (y) and weight percent binder (x). This surface allows visualization of the behavior of a function representing some key aspects of the data. For example, the data suggests that a maximum strength response exists between 0 and 20 weight percent polymer and at a mid-pyrolysis temperature. The strength slope appears steeper towards higher temperatures than lower ones. Such response surface modeling can be used to make modifications of the disclosed embodiments.

Although we have emphasized the utility of the invention for manufacturing dental restorations, the invention is by no means limited to such utility but has general application to any instance where near net shape products are required. Thus, the invention may be used to make other prosthetic devices, as mentioned above for humans or animals.

The teachings of the invention may also be employed whenever low cost, low temperature ceramic composite materials are desired, such as substitutes for metal and other parts for aircraft, land, water or underwater vehicles, missiles, rockets, and other projectiles where near net shape is a requirement of the application.

Although we have disclosed methyl methacrylate as a preferable non-ceramic firing infiltrant, a large class of other monomers, oligomers and polymers are suitable as infiltrants, such as acrylics, styrenics and other vinyls, epoxies, urethanes, polyesters, polycarbonates, polyamides, radiopaque polymers and biomaterials. Specific examples include the following compounds: acenaphthylene,