

matrix. Table I lists the physical characteristics of glasses suitable for this invention.

TABLE I

Glass	Density (g/cc)	Coeff. of Expansion (10^{-7} per °C.)	Dielect. Const.	Modulus		Softening point (°C.)	Working Temp (°C.)
				GPa	10^6 psi		
Corning 1723 aluminosilicate glass	2.64	46	6.3	69	10	900	1168
Corning 7740 Borosilicate glass	2.23	33	4.6	64	9	821	1252
Corning 7913	2.18	7.5	3.8	69	10	1530	—

The glass chosen must have a suitable dielectric constant if it is to be used for radar or other electromagnetic window applications. On this basis, the 96% silica and borosilicate glass are outstanding. Additionally, the glass chosen must be as resistant as possible to laser damage.

The composite material of this invention can be made by stacking together a plurality of the precursor sheets, each of which consists of an appropriate mixture of silicon nitride whiskers, glass frit and a temporary binder, and hot-pressing the sheets together at a temperature and pressure sufficient to cause the glass to flow and form a void-free matrix throughout which are dispersed the silicon nitride whiskers. The temporary binder may be burnt out during the hot-pressing operation in some embodiments of this invention, however it has generally been found preferable to burn out the binder in a heat treatment in air prior to hot pressing.

The conditions of hot-pressing are determined by the physical properties of the glass. In general, the temperature will range from about 50° C. to about 300° C. above the normal softening point of the glass, the pressure will range from about 500 to 4000 psi and pressing time will range from about 5 min to 6 hours. The hot-pressing may be carried out under vacuum or under a suitable inert atmosphere such as argon, using graphite or metal dies coated with a suitable high temperature release agent such as, for example, colloidal boron nitride.

The following examples illustrate the invention:

EXAMPLE I

Small clumps of matted whiskers weighing about 1 gram were pulled from mats of silicon nitride whiskers obtained from Versar Incorporated and added to about 500 ml of water in a blender jar. The mixture was blended about 3 minutes at the low speed setting of a Waring Model 5011 blender. Ten such batches were combined, after blending, in a plastic pail and 400 ml conc. HF was added thereto. The mixture was stirred, then allowed to settle for about 1 hour. The liquid above the settled-out whiskers, containing the finest whiskers in suspension, was drawn off using suction from an aspirator. The settled-out whiskers were washed by adding about 5-l. of distilled water, stirring the mixture and allowing the mixture to settle for about an hour. The liquid above the settled-out whiskers was drawn off, as above. The washing procedure was repeated for a total of four washings.

The whisker mass was then collected using an aspirator to draw the whiskers and water remaining in the pail through a millipore filter. The whisker cake was then dried.

EXAMPLE II

15 21.6 g of the whiskers obtained in Example I were dispersed in 900 ml of distilled water. To this were added 32.6 g of Corning 1723 glass (approx. 325 mesh) frit and 22 g of Rhoplex Grade AC-33 acrylic latex. The mixture was thoroughly stirred then poured into a pan lined with a polyester release film. The water was evaporated off, yielding a paper-like precursor sheet.

Three-inch squares were cut from the precursor sheet. A plurality of such squares were stacked in a stainless steel jig. The jig containing the stacked squares was heated to 500° C. in an air atmosphere for 1.5 hours to decompose the temporary binder. The temperature was thereafter increased to 750° C. for 30 min. to at least partially sinter the glass frit to allow transfer of the squares to a hot-pressing jig without disturbing the distribution of whiskers and glass. After cooling to room temperature, the stack of squares was transferred to a graphite hot-pressing mold. The mold was placed in a hot press, heated to 1100° C., pressed at 1000 psi for 5 minutes, then cooled to room temperature.

The resulting hot-pressed composite plate was ground and cut into 0.10" × 0.20" × 3.0" test bars. These bars were tested in three point flexure at a span:depth ratio of 25:1 at a loading rate of 0.005 inch per minute. These composites exhibited a mean flexural strength of 21 ksi. In contrast, unreinforced Type 1723 glass has a mean flexural strength below 10 ksi.

EXAMPLE III

Small clumps of matted whiskers were pulled from mats of silicon nitride whiskers obtained from Versar Incorporated, weighed and slurried with Corning 1723 glass frit and Rhoplex acrylic latex, then formed into paper-like precursor sheet as described in Example II. These whiskers were not treated with HF as described in Example I. The thus-formed precursor sheet was processed into a hot-pressed composite plate, which was ground and cut into test bars, as described in Example II. This composite exhibited a mean flexural strength of 12 ksi.

Microphotographs of the composite structure of this Example exhibited good "wetting" of the fiber clumps by the 1723 matrix glass, however, the distribution of the fibers was clumpy. In contrast, microphotographs of the composite structure of Example II exhibited a reasonably planar isotropic distribution of the fibers as well as good "wetting" of the fibers. The homogeneity of the latter composite is reflected in the higher strength of this composite as compared to the strength of the composite of Example III.

Various modifications may be made in the present invention without departing from the spirit of the invention or the scope of the appended claims.

I claim: