

**REACTION INHIBITED-SILICON CARBIDE  
FIBER REINFORCED HIGH TEMPERATURE  
GLASS-CERAMIC COMPOSITES**

**TECHNICAL FIELD**

The field of art to which this invention pertains is fiber reinforced composites.

**BACKGROUND ART**

Because of the scarcity and increasing expense of many conventional high temperature structural metals, increased attention has focused on non-metal containing composites as replacement for conventional high temperature metal-containing materials. Use of metal replacement, high strength fiber reinforced resin and even high strength fiber reinforced metal matrix composites has progressed to the point of commercial acceptance in products ranging from sporting goods to advanced jet aircraft components. One of the big problems with these composites, however, has been their maximum use temperature.

Ceramic, glass, and glass-ceramic bodies are known to the art which can be employed in high temperature applications. Unfortunately, however, those bodies frequently lack the mechanical strength desired and are invariably deficient in toughness and impact resistance. This situation has given rise to the preparation of composite bodies consisting of a matrix of ceramic, glass, or glass-ceramic material with inorganic fibers dispersed in continuous or discontinuous fashion therewithin.

Nevertheless, while such composites, for example, graphite fiber reinforced glass and alumina fiber reinforced glass, can be utilized at higher use temperatures than conventional high temperature structural metals, there is still much room for improvement. To illustrate, while the graphite fiber reinforced glass composite demonstrates high levels of strength, fatigue resistance, and fracture toughness, it is also susceptible to detrimental fiber oxidation at elevated temperatures. And while composites such as alumina fiber reinforced glass are oxidatively stable at high temperatures, the overall strength and toughness levels obtainable with these composites are less than those possible with a graphite reinforced glass system, for example. Similarly, high strength and toughness properties have been obtainable with silicon carbide fiber reinforced glass composites (note U.S. Pat. No. 4,314,852) and silicon carbide fiber reinforced ceramic composites (note U.S. Pat. No. 4,324,843).

Although glass-ceramic bodies customarily exhibit greater refractoriness and strength than their precursor glasses, there has been the desire to impart even higher mechanical strengths thereto. However, silicon carbide fibers have demonstrated a tendency to react with glass-ceramic matrices at high temperatures, which phenomenon has been a limiting factor in their utility as reinforcing elements. Accordingly, what is needed in the art is a composite with high strength, high fracture toughness, and oxidation stability at high temperatures.

**DISCLOSURE OF INVENTION**

The present invention is directed to a solution to the high temperature strength, fracture toughness, and oxidation stability problems which exist with composites of the prior art and comprises silicon carbide fiber reinforced glass-ceramic composites, wherein said glass-ceramic matrices have compositions within the base

$\text{Li}_2\text{O}-\text{A}_2\text{O}_3-\text{SiO}_2$  system and contain  $\text{As}_2\text{O}_3$  and  $\text{Nb}_2\text{O}_5$  and/or  $\text{Ta}_2\text{O}_5$ .

The high strength composites according to the present invention comprise silicon carbide fibers in a glass-ceramic matrix wherein the tantalum and/or niobium ions in the matrix react during composite fabrication with the surface of the silicon carbide fibers to form reaction-inhibiting, diffusion barrier layers thereon. The fibers can be laid in continuous or discontinuous fashion in the matrix and result in composites with high strength (e.g., much greater than the matrix itself) and oxidative stability even at high temperatures (e.g., in excess of  $1000^\circ\text{C}$ . and, preferably, up to  $1200^\circ\text{C}$ .) for prolonged periods of time.

The foregoing, and other features and advantages of the present invention, will become more apparent from the following description and accompanying drawing.

**BRIEF DESCRIPTION OF DRAWING**

The Figure shows a fiber reinforced composite according to the present invention.

**BEST MODE FOR CARRYING OUT THE  
INVENTION**

A glass, which can be converted to a glass-ceramic, is the ideal matrix material to form the composites of the present invention. During composite densification the matrix is retained in the glassy state, thus avoiding fiber damage and promoting densification under low applied pressure. After densification to the desired fiber plus matrix configuration, the glassy matrix can be converted to the crystalline state, the degree and extent of crystallization being controlled by the matrix composition and heat treatment schedule employed. A wide variety of glasses could be used in this manner; however, limitation on the amount and activity of titanium present in the glass is preferred since the titanium appears to compete with the niobium and tantalum in reacting with the silicon carbide fibers, resulting in a decrease in composite properties. Thus, titanium is capable of reacting with the silicon carbide fiber to form titanium silicides around the silicon carbide fiber, which severely degrade the fiber strength and, as a consequence, the strength and fracture toughness of the composite are greatly lowered. Accordingly, if titania nucleating agents are used, they are preferably inactivated or kept below one percent by weight. This can be accomplished by simply substituting another nucleating agent such as zirconia for the conventional titania or adding an agent to mask the reactivity of the titania toward the silicon carbide fiber. However, in any case it is important to either eliminate or mask the effects of the titania on the silicon carbide fiber to attain a composite with the improved properties disclosed.

In addition to eliminating or masking the effect of titanium on the silicon carbide fibers, to obtain composites with the improved properties disclosed, the addition of either niobium or tantalum ions to the glass-ceramic is necessary. These ions promote the formation of either a niobium carbide or tantalum carbide reaction barrier layer around the silicon carbide fibers during the composite fabrication step. This reaction barrier prevents any interaction between the glass-ceramic matrix and the silicon carbide fibers at elevated temperatures (about  $900^\circ$  to about  $1200^\circ\text{C}$ .) in air or other oxidizing environment.