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Monazite — a High Temperature Ceramic Interface

Over the past 30 years damage tolerant ceramic composites have been developed by introducing weak interfaces to stop crack growth through the entire brittle body. The best known of these man-made composites is the fiberglass/epoxy material, which has innumerable applications at ambient temperatures. Attempts to make composites for high temperature applications have been limited. In general, the high temperature composites are made up of a hard refractory brittle matrix (e.g. silicon carbide, silicon nitride, or glass), a reinforcement (e.g. carbon, or silicon carbide), and a weak bonding interface material (e.g. graphite or boron nitride). These composites have high strength and fail in a non-brittle manner, but the interface materials are not stable at high temperatures under oxidizing conditions. This is unfortunate, since the high temperature properties of these ceramic composites are the most likely candidates for structural components in advanced engines and in power generation systems.

Recent work by P.E.D. Morgan and D. B. Marshall of the Rockwell International Science Center, Thousand Oaks, California found that LaPO_4 , which has the monoclinic monazite (a rare earth phosphate) type structure, works well as a weakly bonded interface for a composite consisting of an alumina matrix and alumina reinforcement. The LaPO_4 and alumina (Al_2O_3) are compatible up to at least 1750°C , and are sufficiently weakly bonded that a crack in LaPO_4 will deflect along an interface with Al_2O_3 rather than across the interface into the Al_2O_3 grain. The authors tested both a simple composite consisting of sapphire (Al_2O_3) fibers in a LaPO_4 matrix, and a more complex laminar composite consisting of alternate layers of Al_2O_3 and LaPO_4 . The materials were sintered in air at 1600°C before testing the crack propagation characteristics of the composites. Still to be demonstrated are the high temperature strength, failure mode, and oxidation resistance of such composites. But the initial tests look encouraging. As the authors point out, from the mineralogy of LaPO_4 , it is compatible with silicates, and so it may also be a useful interface material for other common refractories such as mullite and cordierite.

Er in Si Incorporated

No! This is not a story about a new company devoted to Er. Instead, it is a brief summary about a clever, and probably inexpensive way of incorporating erbium into silicon. The main interest in Er-doped silicon is its light emission at $1.54\mu\text{m}$ for use in optoelectronic devices (see **RIC Insight 6**, [4] April 1993). The usual method for incorporating erbium into silicon is by ion implantation followed by thermal annealing. This requires a high voltage implanter (e.g. 500 keV) and a long implant time to attain a sufficiently high erbium volume density ($\sim 10^{20}/\text{cm}^3$) for strong luminescence. Recently, a team of Japanese scientists, headed by T. Kimura (from the University of Electro-Communications, the University of Tokyo, and Fujikura Ltd., all from Tokyo), developed

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an electrochemical method of incorporating erbium into the silicon. Initially, the *p*-type silicon surface is anodically etched in HF solution to make the surface porous. This treated surface is then immersed in an ErCl_3 ethanol solution and a negative bias is applied drawing the Er^{3+} ions into the fine pores by the electric field. After thermal annealing at $\sim 1300^\circ\text{C}$ in an O_2 -Ar atmosphere, the samples show a sharp and intense photoluminescence at $1.54\mu\text{m}$ upon excitation with an Argon laser. The 1300°C oxygen anneal is essential to form Er-O complexes which are necessary for optical activation, as has been reported earlier (see **RIC Insight 5** [12] December 1992). These results were published in **Appl. Phys. Lett.** **65**, 983-985 (1994).

New Class of Nd Lasers

Scientists at Lawrence Livermore National Laboratory (Livermore, California) and the University of Central Florida (Orlando) have successfully grown Nd-doped $\text{Sr}_5(\text{VO}_4)_3\text{Cl}$ single crystals and have demonstrated a laser emission at $1.065\mu\text{m}$. They also described the lasing action of Nd-doped $\text{Ca}_5(\text{VO}_4)_3\text{F}$ and $\text{Sr}_5(\text{VO}_5)_3\text{F}$ crystals. The team of scientists, headed by L. D. DeLoach, believe these vanadate apatite crystals will become an important host material for low-threshold diode-pumped Nd laser systems. According to DeLoach *et al.* [**Appl. Phys. Lett.** **65**, 1208-1210 (1994)], high quality crystals can be readily grown by the Czochralski technique. Of the eight halovanadates studied, the best material is Nd-doped $\text{Sr}_5(\text{VO}_4)_3\text{F}$ with a slope efficiency of 66%, followed by Nd-doped $\text{Sr}_5(\text{VO}_4)_3\text{Cl}$ with a 52% slope efficiency. They believe that efficiency of the latter can be improved if higher quality crystals can be obtained.

Yttria Coated AlN

Aluminum nitride (AlN) is used in electronic substrates and packages for semiconductor devices which generate a large amount of heat because of its good thermal conductivity, high electrical resistivity and nontoxicity. Oxygen is known to have an adverse affect on the thermal conductivity of AlN, and in order to keep the oxygen content low, Y_2O_3 or CaO are used as sintering aids to make AlN ceramics. The Y_2O_3 (CaO) react with the oxide layer, which exists on the AlN surface, during the sintering process, and this prevents the oxygen from diffusing into the AlN grains. Normally the AlN and Y_2O_3 (or CaO) powders are mechanically mixed, but it has been found that the more uniformly the additive(s) are distributed the more effectively the Y_2O_3 will react with the surface oxide layer, resulting in a high thermal conductivity. Recently, W.-J. Kim and co-workers have described a method for coating a Y_2O_3 precursor on the AlN powder by *in situ* precipitation in a nonaqueous system [**J. Mater. Sci. Lett.** **13**, 1349-1351 (1994)]. The procedure consisted of ball milling the AlN powder in a solution of $\text{Y}(\text{NO}_3)_3$ in dry isopropanol for 6 hours. This slurry was treated with ammoniated isopropanol, filtered and washed, and then dried at 60° before sintering at 1750°C or higher. The resultant composite consisted of small, uniformly distributed Y_2O_3 particles in the sintered AlN matrix, which had essentially no voids. The mechanically milled material, on the other hand, consisted of a wide range of particle sizes of Y_2O_3 , non-uniformly distributed in the AlN matrix, which also contained many voids. Furthermore, the thermal conductivity of the former was 10% larger than that of the mechanical milled composite. It appears that the Koreans have developed a process for improving AlN for substrates and coatings in semi-conductor devices.

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