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Fracture toughness of hydroxide catalysis bonds between silicon carbide and Zerodur low thermal expansion glass-ceramic

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HIGHLIGHTS

- Fracture toughness of the HCB bonds was found to be higher than 1 MPa $m^{1/2}$.
- A surface pre-treatment procedure was introduced for SiC substrates.
- This eliminates the need for thermally oxidizing the SiC surface before bonding.

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ABSTRACT

In many optical and precision engineering applications, low thermal distortion materials need to be bonded together reliably. Since high temperature bonding process ultimately introduce stresses in the bond, rendering it dimensionally instable, room temperature or near room temperature processes are preferred. Low thermal distortion materials such as silicon carbide and low thermal expansion glass ceramics are bonded at room temperature using hydroxide catalysis bonding with a silicate bonding material. The bonding procedure is explained and fracture toughness results are presented for SiC–SiC, Zerodur–Zerodur and SiC–Zerodur bonds. A surface treatment technique for hydrating the SiC surface is presented, which eliminates the need for pre-oxidized SiC surfaces when using HCB bonding. The bonds between surface treated bare SiC surfaces and thermally oxidized SiC surfaces are found to have comparable fracture toughness.

1. Introduction

In precision metrology and precision manufacturing, such as space instrumentation and various semiconductor manufacturing disciplines, dimensional stability is of paramount importance for the performance of the equipment. For instance in the GAIA space mission the positional stability of the optical components relative to each other is required to be $1.9 \cdot 10^{-13}$ m over a period of 6 h [1]. The most important distorting factors are stress and temperature fluctuations, causing distortions in the materials.

Therefore the choice of materials for manufacturing these components is limited to materials with a high elastic modulus and creep resistance and a low coefficient of thermal expansion α, or low thermal distortion ratio α/κ, with κ being the thermal conductivity of the material. Such materials include silicon carbide and low thermal expansion glass ceramics, such as Zerodur (Schott Glass). Extremely high tolerance optical instruments can be made from these materials [2–4]. Silicon carbide has a higher thermal expansion but a comparable thermal distortion, due to its high thermal conductivity. It also has an exceptionally high modulus, making it resistant to mechanical deformations. Furthermore, SiC is tougher than low-α glass ceramics (see Table 2), allowing for lighter designs, which is especially important for space instrumentation. This has led to SiC designs being implemented in the instrumentation in several earth and space based metrology systems where extreme precision is needed [3–5]. To avoid the extreme cost and manufacturability challenges of forming a functional part from monolithic blocks of material, several parts may need to be bonded to form an assembly.

Conventional methods for SiC bonding such as brazing [3,4,6,7] and glass-ceramic bonding such as glass frit bonding [4,8] are high temperature processes. Due to the difference in α between the bond material and the SiC substrate, large residual stresses are formed in the bond upon cooling. These stresses can slowly relax during the

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operational lifetime, which may the compromise dimensional stability of the part. Furthermore, these high temperature processes are completely impractical for bonding different materials because of the differences in thermal expansion coefficients.

Low temperature epoxy joining is sometimes used for bonding optical components, but has several drawbacks. These drawbacks include limited control of the bond thickness and high thermal expansion [9]. Out-gassing can be a problem for optical components, epoxies are prone to moisture uptake [10] and properties can change over time, especially under influence of UV light. Recently, considerable interest has been shown in hydroxide catalysis bonding for low thermal distortion materials [1,11–14], in particular for silica components. These thin bonds are extremely dimensionally stable [15,16]. In Fig. 1, a comparison between the dimensional stability in a single dimension (dl) under influence of stress (σ) and temperature fluctuations (dT) is made for epoxy, glass frit and silicate type HCB type bonds. The dimensional stability under these loads depends on material parameters and geometry. Typical values for these bonds are listed in Table 1.

As can be seen in Fig. 1, the epoxy bond will not meet GAIA requirements unless the temperature is kept essentially constant and the assembly is completely stress free. Even then effects such as UV light degradation and moisture absorption must still be negated. The glass frit bond will need an extremely well controlled atmosphere (dT < 0.001 °C). Also the high temperature processing may cause slow relaxation of residual stresses and therefore dimensional instability over longer periods of time. The hydroxide catalysis bond is the only bond which is dimensionally stable enough to meet GAIA requirements [1] as described above for reasonable temperature fluctuations (<0.5 °C) and stresses (several 10's of kPa). It has therefore been recognized as an extremely stable bond [9,13,15].

Hydroxide catalysis bonding between silica parts typically consists of three phases after the bonding surfaces are mated. The first phase consists of etching of the SiO_2 surface and formation of silicate ions (see equation (1))

$$SiO_2 + OH^- + 2H_2O \rightarrow Si(OH)^-{}_5 \quad (1)$$

Table 1
Material properties and geometry of typical bonding materials for bonding low thermal distortion materials.

Material	Bond thickness [μm]	E [GPa]	α (20 °C) [10^{-6}/K]	$T_{bonding}$ [°C]
Epoxy	20	1	45	≥RT
Glass frit (Schott G017-339)	10	60	5	>400
HCB/silicate [15]	0.1	8	5	≥RT

The etching reduces with reducing pH of the solution and siloxane chains are formed:

$$2Si(OH)^-{}_5 \rightarrow (HO)_3SiOSi(OH)_3 + 2OH^- + 2H_2O \quad (2)$$

The siloxane chains form a solid bond between the surfaces. Finally dehydration occurs as water evaporates at the edges of the bond. This is a slow process at room temperature. When oxide materials other than pure silica are to be bonded, which cannot form a silicate-like network from etching the bonding surface alone, the bonding process needs to be facilitated by an existing silicate-like network in the solution. A sodium silicate bonding solution (commonly available in 14% NaOH and 27% SiO_2 with a pH of around 11) can be employed the formation of the silicate-like network between other oxides [17] and even other materials with a natural oxide surface [18] including SiC [19]. Different strength measurement methods have led to a wide variety of strengths being reported for HCB or silicate bonds, though little is known about the fracture toughness of the bond. The fracture toughness is important for shock loads, which can occur in during launch and in space. For optimal strength of the bond, the bonding surfaces need to be highly hydrophilic. In previous work with SiC–SiC [1] and Si–Si [13] HCB bonding, a layer of thermal SiO_2 was grown on the SiC or Si surface, to facilitate hydrophilic treatment before bonding and increase the effect of the etching step in the HCB sequence. However, the oxidation process occurs at a fast enough rate only at high temperatures (>1000 °C) and risk of non-uniform coating and

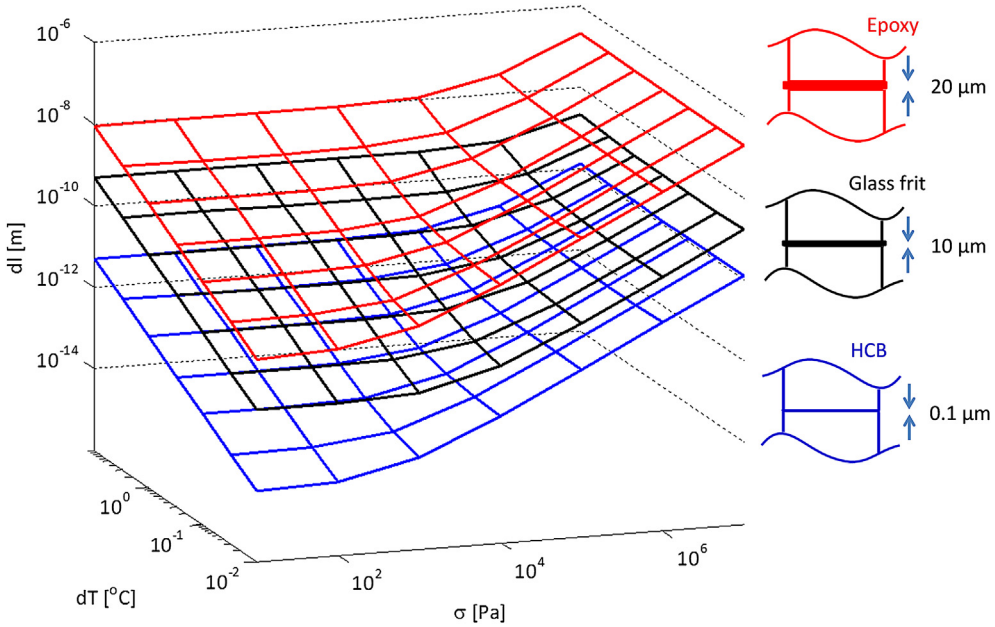

Fig. 1. Length change, dl as a measure of dimensional stability for typical epoxy (red), glass frit (black) and HCB (blue) bonds under varying temperature (dT) and mechanical stress (σ). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)