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BOND FRACTURE STRENGTH IN CERAMIC-TO-METAL JOINTS

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INTRODUCTION

Ceramic to metal joints are used in various areas of ambient and high temperature technology. Examples are metal films on a ceramic substrate in hybrid microelectronics, junctions between electrodes and solid state electrolytes and materials transitions in bone and dental substitutes. Ceramic to metal joints are also of interest for combinations of silicon nitride discs with metal shafts in advanced turbine engines.

Investigations into the bond strength of the joints are of vital importance for assuring the quality of the material. K_{IC}-factors are supposed to give a more sensitive and direct measure of the joint strength than conventional bond strength data.

The present paper reports some results of investigations on the fracture bond strength of a solid state bonded $Al_2O_3/Nb/Al_2O_3$-joint. This composite is used as a model combination without intermediate layers between metal and ceramic. It shows a very good chemical compatibility and a favourable matching of the expansion coefficients. For comparison, fracture resistance values of bulk Si_3N_4 and Al_2O_3, and of Si_3N_4/Zr-joints with intermediate reaction layers were measured.

MATERIALS AND MANUFACTURING PROCESS

The solid state bonded specimens consisted of two sections each of bulk polycrystalline alumina connected by a high purity niobium foil. These combinations were welded together in a high vacuum of $1.3 \cdot 10^{-4}$ Pa by induction heating at temperatures > 1670 K. Likewise hot pressed Si_3N_4 parts were welded together with a Zr-foil of 0.5 mm thickness at 1570 K. The dimensions of the interfacial welded area were 18 x 11 mm, and the length of the welded specimen was 60 mm. A welded specimen was used to obtain four or five bond test specimens with dimensions 7 x 3.5 x 60 mm (Figure 1).

BOND STRENGTH CHARACTERIZATION

The formalism of linear fracture mechanism is valid primarily only for isotropic materials. But it is possible to describe the behaviour of anisotropic materials in like manner [1,2,3]. The same formalism may be applied to brittle orthotropic material combinations, if the intermediate metal layer is thin and the crack runs into the region of the interface parallel to the layer. Therefore, the practice of fracture mechanics testing of ceramics can be applied to the evaluation of the bond strength.

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The bond strength of the joint is then described by analogous K_{IC}-factors which we call K_{ICV}-factors.

For determining K_{ICV}-factors notches (saw cuts) of radius $\rho \leq 50/\mu m$ and depth a were introduced into the metal to ceramic interface. They were thought to be an equivalent to zero volume cracks [4]. The K_{ICV}-factors were evaluated using the well-known formula of Gross and Srawley [5] for isotropic four-point bending specimens (Figure 1).

BOND STRENGTH AND JOINT PROPERTIES

Previous results have shown that welding parameters like temperature and bonding pressure do not seem to influence the K_{ICV}-factor to a measurable degree, if a maximum matching of the contacting surfaces exists [6]. Generally, there are three types of fracture paths in solid state bonded metal to ceramic joints [7]. In composites with fine grained high density alumina the crack extends exactly along the metal to ceramic interface. Joints with a thermoshocked or a very porous ceramic show a crack path within the ceramic near the interface. In joints in which the metal part is seriously embrittled by chemical reactions with the ceramic the fracture is inside the metal foil. As revealed by load deflection curves and illustrated by SEM pictures, the joints fracture in a brittle manner.

RESULTS AND DISCUSSION

In this paper some data on the influence of microstructure, environment, and test temperature on the bond strength K_{ICV} of solid state bonded joints are listed. They characterize the applicability of the fracture mechanics test concept.

The effects of average grain size, percent porosity (density), and impurity content of the ceramic on the K_{ICV}-factors are summarized in Table 1. For comparison a single K_{ICV} value of a Si_3N_4/Zr-joint is given. For low densities of the alumina components the K_{ICV} data are comparatively high exceeding the K_{ICV}-values by a factor of two or three in the case of alumina VIII. This is because of an excellent matching of the surfaces. In contrast, for the highest K_{IC}-data of the ceramic (alumina I,II) the corresponding K_{ICV}-values are zero. Obviously this is a result of the inhomogenity of the micro-structure consisting of small and very large grain sizes. The high K_{ICV}-value with alumina III is probably a consequence of the homogeneous fine grained microstructure.

As shown in Table 2 there is a pronounced influence of various environments, characterized by their polarity E_T [8,9], on the K_{ICV} and K_{IC} data. The crack extends exactly in the metal to ceramic interface. The decrease of bond strength, and of the also measured bulk alumina fracture toughness, with increasing polarity is a function of the silica content of alumina. This environmental behaviour affects the life time of composite and ceramic structures. Because of this influence attention should be payed to the testing conditions of the bond strength measurements.

Table 3 reveals a considerable bond strength of solid state bonded Nb/Al_2O_3 joints up to 1470 K [10]. For this material combination the same fracture paths were observed at ambient and high temperatures. The crack runs precisely between the two materials. Previous measurements with bulk alumina [11] have shown that its decrease in fracture toughness with temperature is more pronounced.

Using a fracture mechanics concept we have tried to overcome the well-known difficulties of conventional bond strength characterization. If it is assumed that the K_{ICV}-factors are materials constants like K_{IC}-factors of isotropic materials, the joint properties may be optimized according to the K_{ICV} values. This concept is suggested to be a useful aid in developing solid state bonded structures for high temperature applications.

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Table 1 K_{IC}-data and microstructure of different Al_2O_3 qualities and Si_3N_4. Corresponding K_{ICV}-data characterizing the bond strength. Testing in air (60% humidity).

Al_2O_3	Purity [w/o]	Density [10^{-3} kg/m^3]	Grain Size d [µm]	K_{IC} [MPa·m$^{1/2}$]	K_{ICV} [MPa·m$^{1/2}$]
I	99.7	3.82	5	4.4	0
II	99.7	3.83	4	4.4	0
III	99.7	3.78	6	3.6	5.6
IV	97.0 SiO_2*	3.70	10	3.5	3.7
V	99.0 MgO*	3.80	15	3.4	3.2
VI	99.0 MgO*	3.84	12	3.4	3.5-2.3
VII	99.7	3.32	2	2.7	3.2
VIII	99.7	2.58	2	0.8	2.5-1.7
Si_3N_4	92.0 MgO, SiO_2	3.18	3	5.5	1.9

K_{ICV}: Al_2O_3/Nb-joints, Al_2O_3 I-VIII, Nb-foil-thickness 0.1 mm

K_{ICV}: Si_3N_4/Zr-joints, Zr-foil-thickness 0.5 mm

Welding temperature: Al_2O_3/Nb, T_W = 1720 - 1870 K

Si_3N_4/Zr, T_W = 1570 K

Welding pressure: P = 1 - 5 · 10^6 Pa; *impurities

Table 2 K_{IC} and K_{ICV} as a function of various environments E_T = Polarity; Nb-foil thickness 0.1 mm.

		1	2	3	4
Medium	E_T kcal/mol	K_{IC} MPa·m$^{1/2}$	K_{IC} MPa·m$^{1/2}$	K_{ICV} MPa·m$^{1/2}$	K_{ICV} MPa·m$^{1/2}$
n-Hexane	30.9	3.9	3.8	3.1	2.4
Silicon oil	35	3.7	3.8	3.0	2.4
Acetone	42.2	3.3	3.7	2.8	2.3
iso-Propanol	48.6		3.7	2.7	2.3
Methanol	55.5	3.2	3.6	2.5	2.2
Water	63.1	3.0	3.2	2.2	2.1
Water*	63.1			2.2	

Column 1 K_{IC}: Al_2O_3 IV as a function of E_T

Column 2 K_{IC}: Al_2O_3 VI as a function of E_T

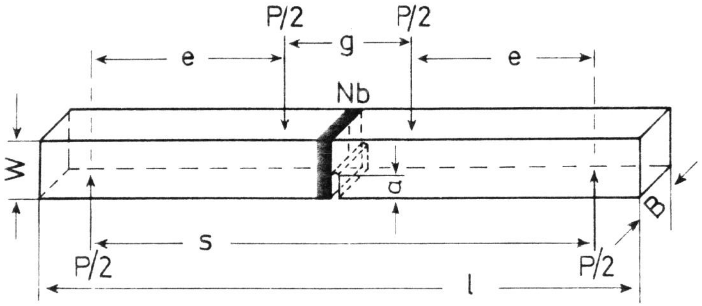
Column 3 K_{ICV}: Al_2O_3 IV/Nb-joint as a function of E_T

Column 4 K_{ICV}: Al_2O_3 VI/Nb-joint as a function of E_T

Water*: after 7 days in water; Al_2O_3 IV, VI see Table 1

Table 3 K_{ICV} as a function of test temperature T_t. Al_2O_3/Nb-joint. Nb-foil thickness is 1.0 mm. Testing in a high vacuum ($1 \cdot 10^{-4}$ Pa).

Joint Parameters	Test Temperature T_t K	K_{ICV} MPa·m$^{1/2}$
Al_2O_3-purity 99.7 w/o	295	2.0
Welding Temperature	1070	2.4
T_W = 2000 K	1270	1.5
Welding Pressure	1470	1.5
P = 5 · 10^6 Pa	1770	0.8



$$K_I = \frac{3P \cdot e\sqrt{a}}{BW^2}\left[1{,}99 - 2{,}47\,(a/W) + 12{,}97\,(a/W)^2 - 23{,}17\,(a/W)^3 + 24{,}80\,(a/W)^4\right]$$

Figure 1 Four point-composite bending specimen with formula for the K_I-factor [5].