

Figure 5 An infra-red spectrum of a mix heated to 800°C.

at 38.82° (2θ) decreases in intensity at 6% NaF and increases with increasing percentage of NaF (12%).

The i.r. band at 1400 cm⁻¹ becomes broader with splitting in the spectrum of the sample containing 12% NaF in comparison to that of samples containing 6% NaF.

For the synthesis of β -Ca₂SiO₄, mixes containing 2% NaF were selected for the present study. The details of heating schedule and analysis of the products are given in Table I. It appears that β -Ca₂SiO₄ is formed even when the sample is fired at 800°C. Formation of spurrite is observed in this product (Figs. 4 and 5). The spurrite XRD lines at 23.4° (2θ) and the carbonate bands at 1400 and 695 cm⁻¹ in the infra-red spectrum can be seen.

From the studies carried out so far, the following observations have been made:

(1) The kinetics are very fast for the reaction to occur at 1000°C, even with a retention time of 30 min or so. Increase of retention time to 90 min increases the yield very little, as was deduced from the XRD line at 37.4° (2θ) for CaO.

(2) Higher temperature 1100, 1200°C etc. did not help the reaction to proceed further, to the contrary, it increases the uncombined CaO content.

(3) Sudden heating of the mix to 1000°C for ½ h appears to have the same result as against the decarbonated mix (800) heated to 1000°C.

Using NaF as mineralizer and adopting rapid heating techniques the kinetics of formation of β -C₂S can be increased substantially. It is, therefore, possible to produce β -Ca₂SiO₄ under controlled conditions with substantial heat economy. However, a certain amount of α' -C₂S phase formation cannot be avoided.

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The effect of ambient atmosphere on the gold-to-alumina solid state reaction bond

In a recent paper by the authors [1], the effect of a number of factors on the bonding of gold to alumina in an air atmosphere was discussed. In that paper, a detailed discussion was given of the preparation of the bonding materials, the actual

bonding process, and the evaluation of the bonds. This paper reports the results of an experimental programme to study the effect of using non-air atmospheres in the formation of this bond. Data are presented for bonds formed in air, oxygen, nitrogen, argon, hydrogen and vacuum.

Briefly, bonds consist of a "sandwich" of two 10 mm diameter rods of Coors polycrystalline

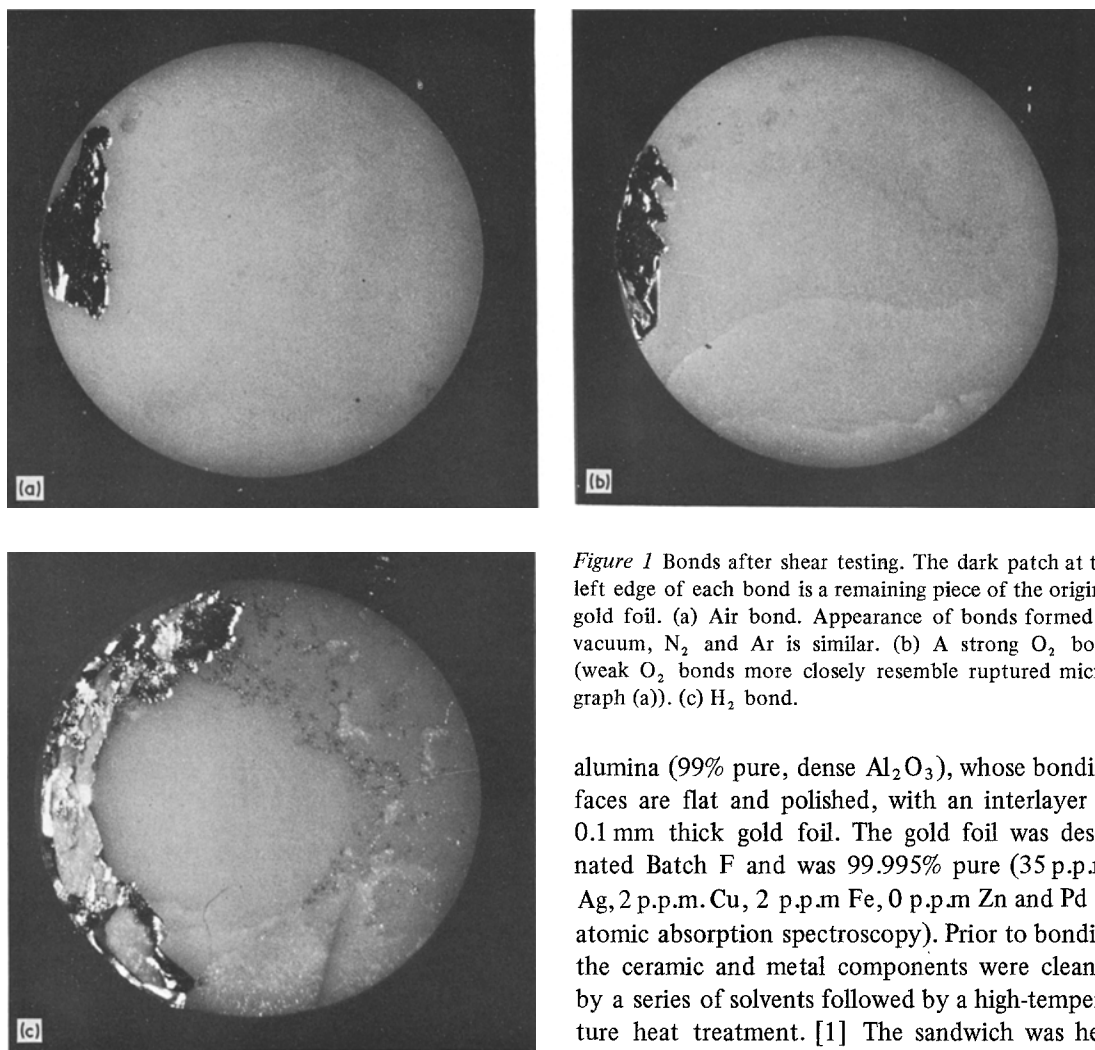


Figure 1 Bonds after shear testing. The dark patch at the left edge of each bond is a remaining piece of the original gold foil. (a) Air bond. Appearance of bonds formed in vacuum, N_2 and Ar is similar. (b) A strong O_2 bond (weak O_2 bonds more closely resemble ruptured micrograph (a)). (c) H_2 bond.

alumina (99% pure, dense Al_2O_3), whose bonding faces are flat and polished, with an interlayer of 0.1 mm thick gold foil. The gold foil was designated Batch F and was 99.995% pure (35 p.p.m. Ag, 2 p.p.m. Cu, 2 p.p.m. Fe, 0 p.p.m. Zn and Pd by atomic absorption spectroscopy). Prior to bonding the ceramic and metal components were cleaned by a series of solvents followed by a high-temperature heat treatment. [1] The sandwich was held under 0.5 MPa contact pressure while being heated in a resistance furnace to a temperature of $1050^\circ C$, and was held at that temperature for 4 to 5 h. The bonds were evaluated by shear testing at room temperature [1].

Control bonds made in an air atmosphere relied on natural convective currents within the furnace to ensure a uniform atmosphere. Non-air atmospheres were controlled by a slow (31 min^{-1}), continuous bleed-in of gas at atmospheric pressure from a gas cylinder through a vacuum-tight tube placed within the furnace. Vacuum was provided by a high-vacuum diffusion pump and oil backing pump. Details of the atmospheres used are given in Table I. Table II tabulates the results of shear tests of the bonds.

TABLE I Details of the atmospheres used in forming reaction bonds. All gases except air were passed through molecular sieves to remove water vapour; oxygen was removed in the case of H_2 by passing it through a catalytic purifier preceding the molecular sieve

Gas	Condition	Pressure (kPa)
Air	Laboratory atmosphere	105
O_2^*	Industrial grade	105
N_2^*	High purity	105
Ar^*	Special grade	105
H_2^*	High purity	105
Vacuum	As measured by Penning gauge	1×10^{-6}

*Supplied by The Commonwealth Industrial Gases Limited, Australia.

TABLE II Shear strengths of bonds formed in various atmospheres

Atmosphere	Number of specimens tested	Average shear strength (MPa)	Coefficient of variation (%)
Air	4	62	8 [†]
O ₂	6	40	48
N ₂	3	26	21
Ar	3	32	21
H ₂	3	27	26
Vacuum	3	33	15

[†]This value is somewhat low, as the strength obtained for one specimen was abnormally low. On observation this was probably due to a defect in the ceramic close to the bond interface. The coefficient of variation is therefore probably much higher.

As can be seen from an examination of Table II, air bonds are significantly stronger than bonds formed in N₂, Ar, H₂ or vacuum. The large scatter in strength of the oxygen bonds is particularly notable.

In addition to the above strength differences the fractured bonds varied in appearance as well. Fig. 1 illustrates typical bond interfaces after shear testing.

When examined at 90× under an optical microscope, fractured bonds formed in air, vacuum, N₂, and Ar show little disruption to the alumina surface with faint infrequent areas of reaction of gold with the alumina surface. The weaker O₂ bonds show this lack of visual interaction as well. The stronger O₂ bonds have large cracks running

across the alumina face and more abundant areas of gold reaction. The H₂ bonds show extensive reaction and disruption of the alumina around the outer edge, along with relatively heavy smears of gold on the surface.

On the basis of both appearance and mechanical strength data, it is concluded that, of the atmospheres explored, air provides the best atmosphere for bonding and certainly the simplest to arrange. In addition to having the greatest mechanical strength, bonds formed in air show little disruption to the alumina interface, an important consideration in most industrial applications. This note reinforces the belief that the reaction bonding process [2] has significant advantages over other ceramic-metal bonding procedures.

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