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Formation of Hercynite (FeAl_2O_4) at Interface of Al_2O_3 /Steel Joint†

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The joining of ceramics to metals has received considerable interest in recent years in connection with the high strength of ceramics. Major methods reported are refractory-metal metallizing, active alloy sealing, oxide-soldering and metal-oxide eutectic

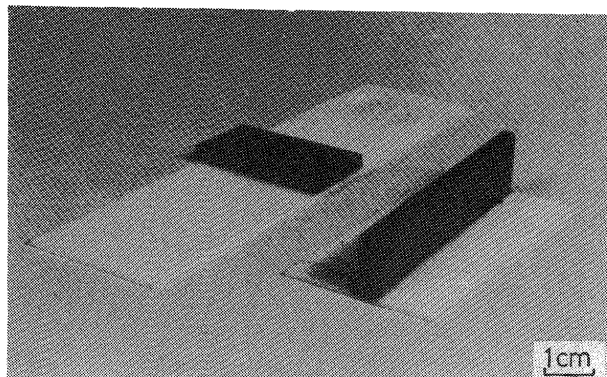


Fig. 1 Al_2O_3 /Steel brazed samples with copper filler metal. (1) alumina butt joint strapped with steel, (2) alumina/steel tee joint.

utilizing¹⁾⁻³⁾. These methods, however, require the complicated process for joining. On the other hand, we succeeded in joining alumina to steel using copper-filler metal in a slightly oxidizing atmosphere. In this work, we will report the intermediary layer compound FeAl_2O_4 formed at a bonding interface between alumina and steel.

The materials used were high purity commercial alumina (95.4%), mild steel (SS41) and copper foil of 0.12 mm in thickness. The joining has been conducted in 10 torr at 1200°C for 30 min. The liquid copper during joining wetted both alumina and steel at 1200°C, and hence a strong bond was formed after cooling down. Various types of joining were produced by using steel and copper filler metals. Alumina butt joint strapped with steel and alumina/steel tee joint are shown in Fig. 1.

In order to characterize the intermediary layer of

Table 1 X-ray diffraction analysis of alumina surface joined in 10 torr at 1200°C for 30 min.

number	$d_{\text{obs.}} (\text{Å})$	Intensity	$\alpha\text{-Al}_2\text{O}_3$ in ASTM card			FeAl_2O_4 in ASTM card		
			$d (\text{Å})$	(hkl)	I/I ₁	$d (\text{Å})$	(hkl)	I/I ₁
1	A 3.440	84	3.479	(012)	75			60
2	2.849	58				2.87	(220)	
3	A 2.529	76	2.552	(104)	90			
4	2.436	100				2.45	(311)	100
5	A 2.362	60	2.379	(110)	40			
6	2.071	100	2.085	(113)	100			
7	2.023	40				2.02	(400)	80
8	A 1.731	49	1.740	(024)	45			
9	1.656	34				1.64	(422)	16
10	A 1.593	68	1.601	(116)	80			
11	1.558	57				1.56	(511)	40
12	1.435	54				1.43	(440)	80
13	A 1.400	42	1.404	(124)	30			
14	A 1.370	68	1.374	(030)	50			

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joint, steel and copper were dissolved from the alumina/steel joint in concentrated hot HNO_3 solution, and X-ray diffraction analysis with $\text{Cu}\cdot\text{K}\alpha$ radiation and X-ray photo-electron spectroscopic examination with $\text{Mg}\cdot\text{K}\alpha$ excitation were conducted to identify the intermediary compound. X-ray diffraction analysis demonstrates the formation of hercynite (FeAl_2O_4) at interface between copper and α -alumina as shown in Table 1. The X-ray reflections were assigned to alumina with rhombohedral structure and hercynite with cubic structure ($a=8.119\text{\AA}$).

The formation of intermediary compound is also known in another system⁹⁾: CuAlO_2 has been found between copper oxide and alumina after reaction at 1150°C in air. In the refractory metal-joining process (moly-manganese), manganese oxide reacts with alumina and forms the intermediary compound $\text{MnO}\cdot\text{Al}_2\text{O}_3$.

Further examination of the intermediary layer compound was carried out by X-ray photoelectron spectrometry. The C1s spectrum (285.00 eV) from the contaminant carbon was used for the calibration of binding energies of electrons. The $\text{Fe}2p_{3/2}$, Al2p and O1s electron spectra are given in Figs. 2-4, respectively.

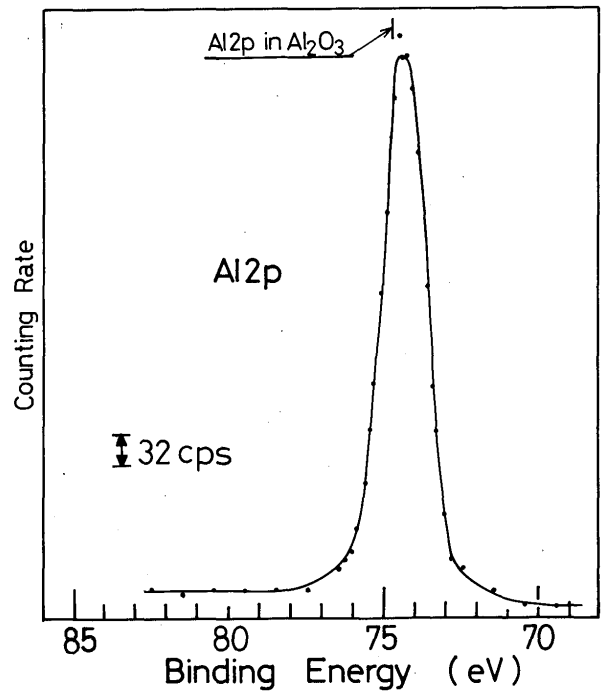


Fig. 3 Al2p spectrum of the revealed surface on alumina, after dissolution of steel and copper from a alumina/steel joint with copper filler.

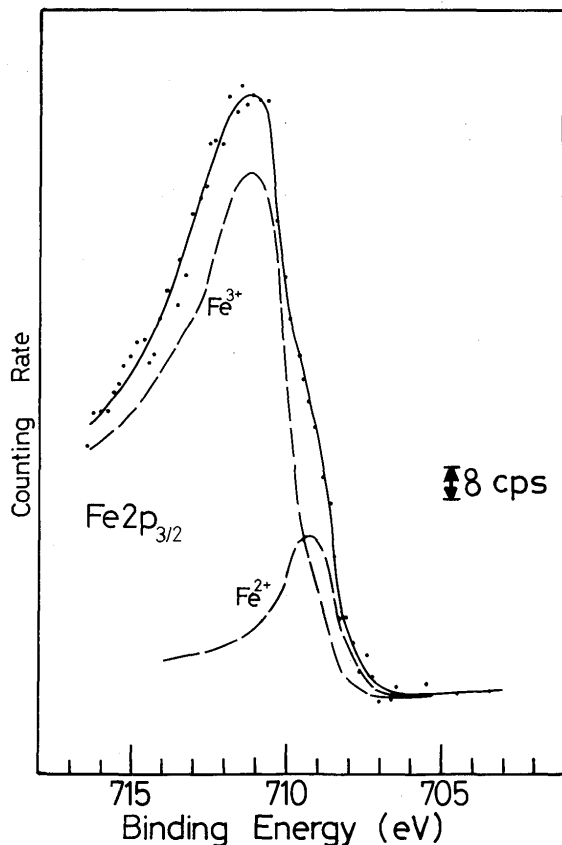


Fig. 2 $\text{Fe}2p_{3/2}$ spectrum of revealed surface on alumina, after dissolution of steel and copper from a alumina/steel joint with copper filler.

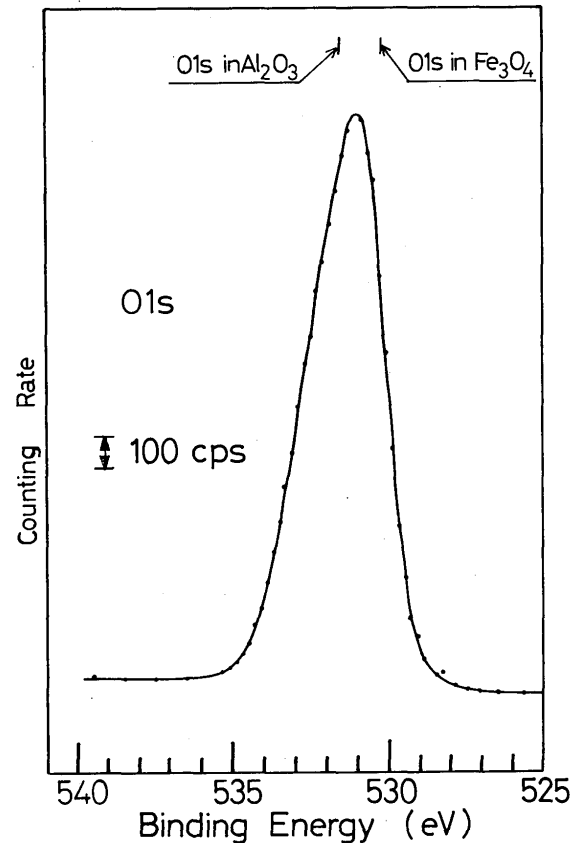


Fig. 4 O1s spectrum of the revealed surface on alumina, after dissolution of steel and copper from a alumina/steel joint with copper filler.

The Fe2p_{3/2} consists of superposed spectra originating from Fe(III) and Fe(II) components⁴. The binding energies of the Fe2p electrons are taken as 711.20 and 709.20 eV for Fe(III) and Fe(II), respectively. The spectrum of Fe(II) can be attributed to hercynite (FeAl₂O₄). The intense spectrum of Fe(III) suggests that the surface of FeAl₂O₄ has been oxidized during specimen preparation, particularly, during boiling in hot concentrated HNO₃. The binding energy of Al2p (74.35 eV) measured is very close to that for Al₂O₃ (74.70 eV). The binding energy of the O1s electrons (531.00 eV) is different from those of Al₂O₃ (531.55 eV) and Fe₃O₄ (530.17 eV), since the binding energy of O1s electrons changes with coexisting cations in the specimen⁵.

It can, therefore, be concluded that the intermediary layer compound FeAl₂O₄ is formed between Al₂O₃ and steel during joining using copper-filler with a consequent strong adhesion.

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