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Author(s)	Okamoto, Ikuo; Naka, Masaaki; Asami, Katsuhiko et al.
Citation	Transactions of JWRI. 1982, 11(1), p. 131-133
Version Type	VoR
URL	<a href="https://doi.org/10.18910/12072">https://doi.org/10.18910/12072</a>
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# Formation of Hercynite ( $\text{FeAl}_2\text{O}_4$ ) at Interface of $\text{Al}_2\text{O}_3$ /Steel Joint†

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KEY WORDS: (Ceramics) (Joining) (Alumina) (Steel) (Hercynite) (X-ray Photoelectron Spectra)

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

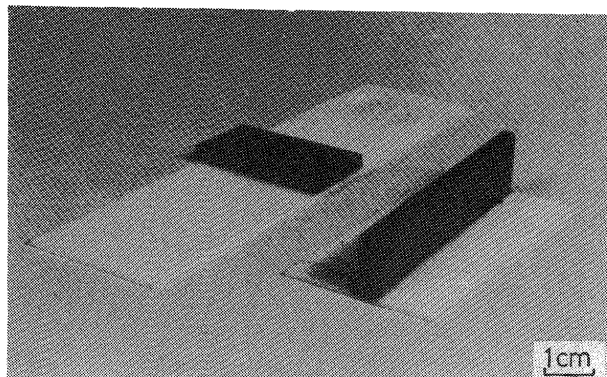


Fig. 1  $\text{Al}_2\text{O}_3$ /Steel brazed samples with copper filler metal. (1) alumina butt joint strapped with steel, (2) alumina/steel tee joint.

utilizing<sup>1)-3)</sup>. 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  $\text{FeAl}_2\text{O}_4$  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			$\text{FeAl}_2\text{O}_4$ in ASTM card		
			$d (\text{Å})$	(hkl)	I/I <sub>1</sub>	$d (\text{Å})$	(hkl)	I/I <sub>1</sub>
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			

† Received on March 31, 1982

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joint, steel and copper were dissolved from the alumina/steel joint in concentrated hot  $\text{HNO}_3$  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 ( $\text{FeAl}_2\text{O}_4$ ) at interface between copper and  $\alpha$ -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 system<sup>9)</sup>:  $\text{CuAlO}_2$  has been found between copper oxide and alumina after reaction at  $1150^\circ\text{C}$  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.

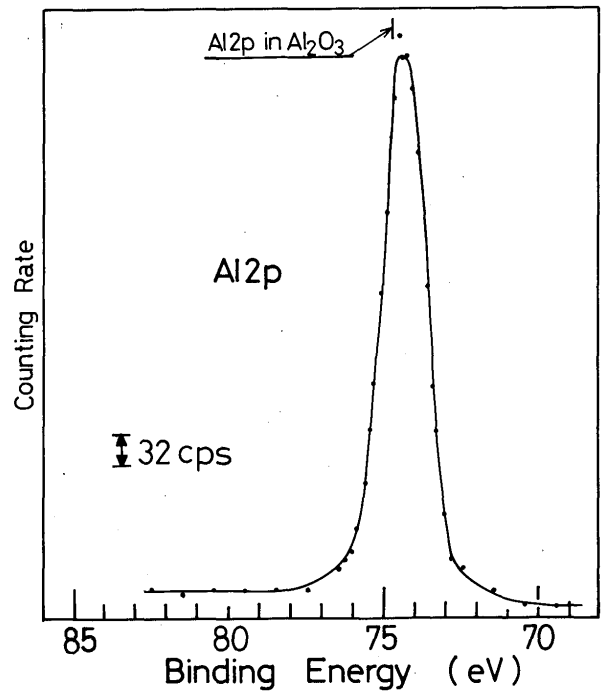


Fig. 3 Al2p spectrum of the revealed surface on alumina, after dissolution of steel and copper from a alumina/steel joint with copper filler.

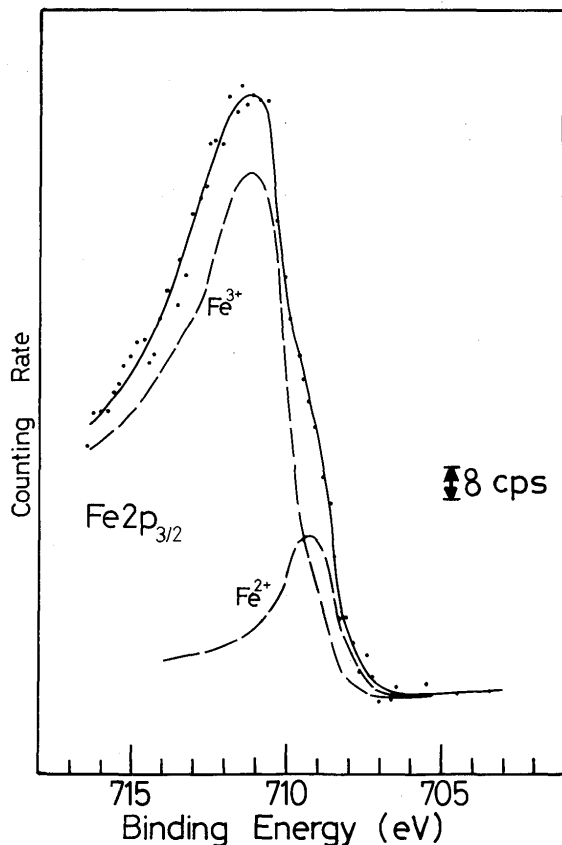


Fig. 2  $\text{Fe}2p_{3/2}$  spectrum of revealed surface on alumina, after dissolution of steel and copper from a alumina/steel joint with copper filler.

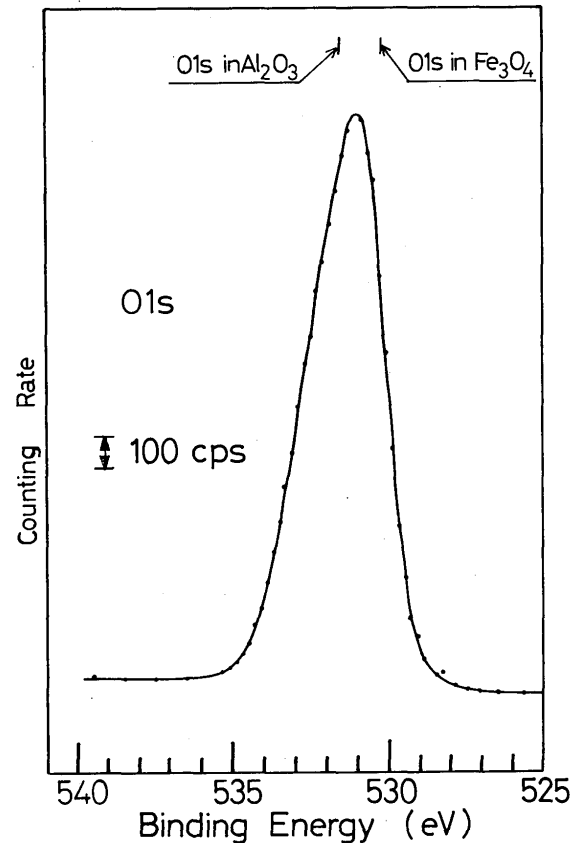


Fig. 4 O1s spectrum of the revealed surface on alumina, after dissolution of steel and copper from a alumina/steel joint with copper filler.

The Fe2p<sub>3/2</sub> consists of superposed spectra originating from Fe(III) and Fe(II) components<sup>4</sup>. 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 (FeAl<sub>2</sub>O<sub>4</sub>). The intense spectrum of Fe(III) suggests that the surface of FeAl<sub>2</sub>O<sub>4</sub> has been oxidized during specimen preparation, particularly, during boiling in hot concentrated HNO<sub>3</sub>. The binding energy of Al2p (74.35 eV) measured is very close to that for Al<sub>2</sub>O<sub>3</sub> (74.70 eV). The binding energy of the O1s electrons (531.00 eV) is different from those of Al<sub>2</sub>O<sub>3</sub> (531.55 eV) and Fe<sub>3</sub>O<sub>4</sub> (530.17 eV), since the binding energy of O1s electrons changes with coexisting cations in the specimen<sup>5</sup>.

It can, therefore, be concluded that the intermediary layer compound FeAl<sub>2</sub>O<sub>4</sub> is formed between Al<sub>2</sub>O<sub>3</sub> and steel during joining using copper-filler with a consequent strong adhesion.

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