

Metal-to-ceramic bonding in $(\text{Al}_2\text{O}_3 + \text{Fe})$ cermet studied by Mössbauer spectroscopy

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Abstract. Although a strong thermo-compression bond is formed between some metals and ceramics, the bonding mechanism is not clearly understood. In the present work ^{57}Fe Mössbauer spectroscopic study of $(\text{Al}_2\text{O}_3 + \text{Fe})$ cermet is carried out to investigate the reason of adherence at the metal-ceramic interface. The isomer shift, quadrupole splitting and linewidths of Mössbauer spectra indicate that a compound of the spinel type $\text{FeO} \cdot \text{Al}_2\text{O}_3$ is formed but the site symmetry around Fe may be locally distorted by a small amount as compared to that for stoichiometric FeAl_2O_4 .

Keywords. Cermet; thermo-compression bonds; Mössbauer spectroscopy; spinel.

1. Introduction

A strong bonding between metals and ceramics is produced by thermal compression. It is of interest to find out why adherence is produced between a non-reactive metal (such as Fe) and a refractory oxide (Al_2O_3) although from a thermodynamic point of view the free energy decrease for metal oxide formation (-58 kcal for FeO) is less than the energy required to reduce the refractory oxide (-125 kcal per oxygen atom for Al_2O_3). The methods used to produce these composites and the earlier investigations have been discussed by Klomp (1970). It was earlier hypothesised that there is first an evaporation of Fe on alumina surface and subsequently a contact between the metal-metal interface which results in bonding. However, Klomp and Lindenhovius (1978) prepared sintered compacts of alumina and iron powders to increase surface area and found no evidence for such a mechanism. The resistivity measured for the composite was same as that for pure alumina indicating no increase in metal-metal contacts. Mössbauer spectroscopy is ideally suited to study the bonding mechanism and an investigation of $(\text{Al}_2\text{O}_3 + \text{Fe})$ system is presented in this work.

2. Experimental procedure

The samples were prepared by sandwiching an alpha iron foil of $10 \mu\text{m}$ thickness between two sintered alumina discs (1 cm diameter \times 0.2 cm thickness). These were then placed under a tightened screw-cap under a pressure of about 0.2 kg/cm^2 and heated in argon atmosphere at 1250 C for 30 minutes (figure 1). This temperature corresponds to $0.9 T_m$ (where T_m is the melting point of Fe) as recommended by Klomps (1970). Mössbauer spectra were recorded for the sample in transmission geometry with a ^{57}Co in rhodium source.

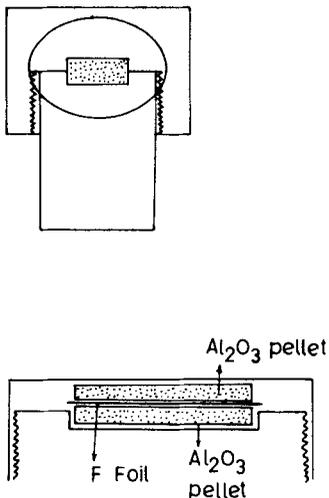


Figure 1. Fe foil sandwiched between two ceramic pellets and pressed in a tightened screw cap. The encircled part is shown in detail in bottom diagram.

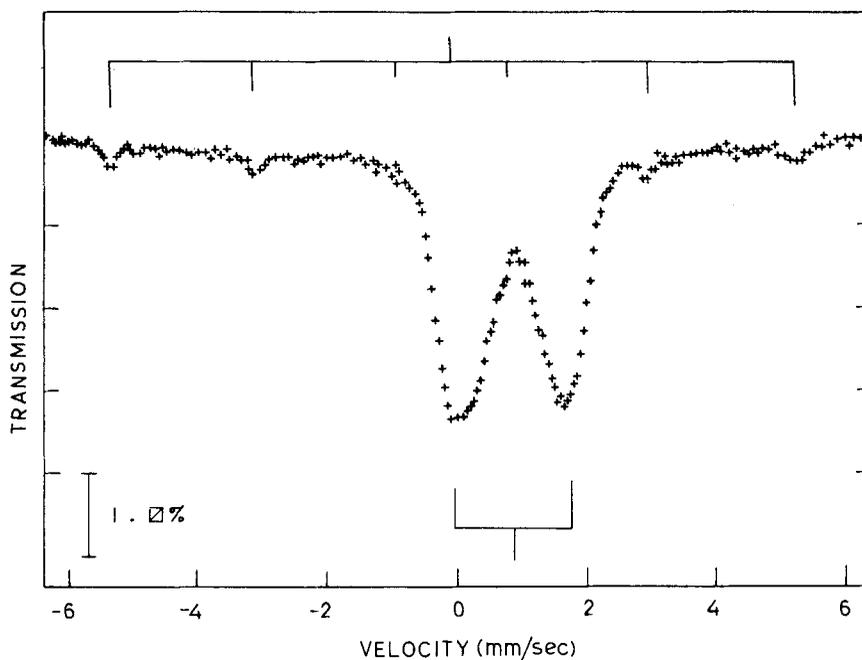


Figure 2. Mössbauer spectrum for the cermet ($\text{Fe} + \text{Al}_2\text{O}_3$) sample. The weak six line spectrum corresponds to α Fe and the absorption in central region is due to a broadened quadrupole split doublet. The position of lines obtained by χ^2 fit is indicated.

3. Results

The Mössbauer spectrum at room temperature, shown in figure 2, consists of a broadened doublet near the centre together with a small absorption in the higher velocity regions. The absorption in the higher velocity regions was identified as that due

Table 1. Comparison of results for $(Al_2O_3 + Fe)$ cermet with data of similar systems

Sample	Isomer shift (mm/sec)	Quadrupole splitting (mm/sec)
$(Al_2O_3 + Fe)$ cermet	0.94	1.78
Tetrahedrally coordinated ferrous halides	0.8 – 1	—
Fe(II) covalent compounds	0.8 – 1.5	—
$Fe_{1-x}O_x$ (wusite)	Site A: 0.91	0.46
$0.9 \leq x \leq 0.93$	Site B: 0.86	0.78
αFe_2O_3	0.4 (SS)	0.12
$FeOAl_2O_3$ spinel	1.08	1.6

to a small amount of unreacted iron by comparison with a standard Fe spectrum. The inner lines were fitted to a doublet and the parameters for the fit are shown in table 1. The linewidths of the component lines of the doublet are large (1 mm/sec). However when an attempt was made to fit to two doublets there was a slight increase in χ^2 .

In table 1 the isomer shifts and quadrupole splittings are compared for various Fe compounds (Greenwood and Gibb 1971). The observed isomer shift for the cermet lies in the range of isomer shift values for Fe^{2+} covalent compounds. Further, both isomer shift and quadrupole splitting values for the cermet lie very close to the ones for the spinel $FeO \cdot Al_2O_3$. It therefore seems likely that bonding takes place by formation of a spinel-type compound at the surface. The compound formed may not be stoichiometric as is suggested by the large line broadening. A small range of quadrupole splitting values would lead to such a broadening of the lines. The coordination symmetry around Fe is therefore not exactly the same as in the spinel structure but slightly distorted, the amount of distortion varying from site to site. There are other evidences for such a compound formation of contacting materials when Nb, Ti, Ta and Zr metals are involved in bonding with ceramics (Buyers 1963).

4. Conclusion

The ^{57}Fe Mössbauer spectroscopy study of metal and ceramic interface in $(Al_2O_3 + Fe)$ cermet shows that (i) there is very little unreacted iron ($< 0.1\%$). This also confirms with the study of Klomp and Lindenhovius (1978) who ruled out bonding by an increase of metal-metal contacts by resistivity measurements. (ii) Bonding takes place by the formation of a compound of the type of $FeAl_2O_4$ spinel at the interface but with slight deviations from coordination symmetry around the Fe atom as compared to that in $FeAl_2O_4$.

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