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Reaction of Zirconia with Carbon Steel in a Vacuum Condition

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Wide applications of zirconia ceramics have been tried in many fields after the production of high strength YTZ was succeeded. However, high strength zirconia-metal joint is required for its more extensive applications. Until now, high strength zirconia-steel joint without any insert material has not been obtained. In the present study, solid state reaction of zirconia to carbon steel in high vacuum conditions was investigated using various spectroscopic methods in order to characterize the interface of the joint.

Joining of zirconia (3 mol% Y_2O_3) to carbon steel (SM-50A) was performed at the temperature of 800°C to 1200°C for 1 hr under a vacuum condition below 1×10^{-5} torr. Square disks of zirconias and carbon steels were used and their dimensions are 8mm X 8mm X 3mm, respectively. Before joining, surfaces of these disks were polished using emery papers and washed in ethanol using ultrasonic waves.

Weak joints were obtained when joining was performed at 1000°C and any joint was not obtained in anothor experimental conditions. However, color of zirconia surface changed in every experiment before and after joining treatment.

Scanning electron micrograph of zirconia-carbon steel joint is shown in **Fig. 1**. Dark region is observed in zirconia near the interface. XPS results are given in **Fig. 2** and **Table 1**. Peaks of O_{1s} and Zr_{3d} photoelectrons are observed at a higher positions than those obtained from normal standard oxides. Fe_{2p} photoelectron peak appears at somewhat lower position than that of hematite[1]. Accordingly, it is suggested that iron is in a intermediate state between trivalent and divalent iron ions, or are in a mixed state of Fe^{3+} and Fe^{2+} ions. ESR result, which is shown in **Fig. 3**, supports the existence of trivalent iron ions in zirconia after reacting to iron. The formation of isolated and dipole-dipole interated ferric ions is also supported from ESR result though the quantity of isorated ferric ions is very low[2]. Furthermore, x-ray diffraction analysis for thin film region identified the formation of magnetite phase at surface layer in zirconia after reacting to carbon steel. Conclusively, iron dissolves into zirconia in the form of trivalent state irrespective of reacting carbon steel with zirconia at a vacuum condition below 1×10^{-5} torr.

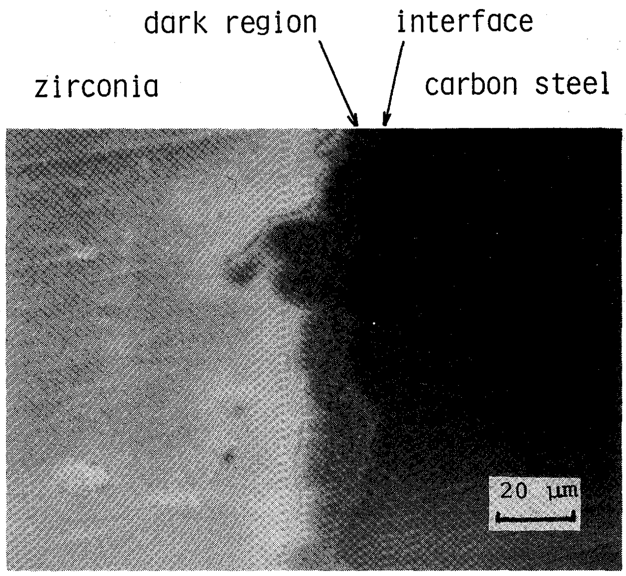

Fig. 1 Scanning electron micrograph of zirconia-carbon steel joint.

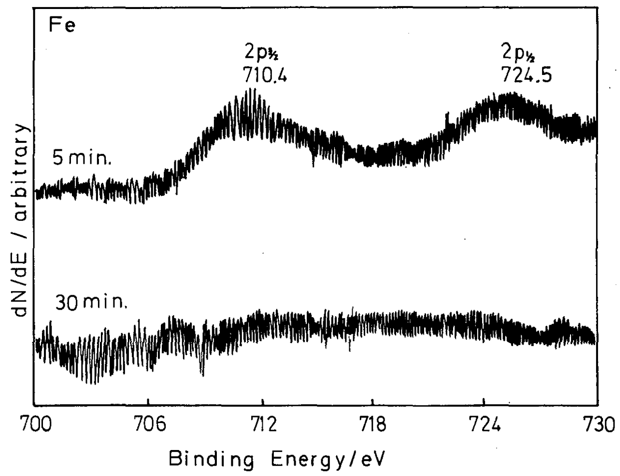

Fig. 2 X-ray photoelectron spectra of $Fe_{2p(3/2)}$ obtained from zirconia surface of zirconia-carbon steel joint.

Anomalous chemical shifts of O_{1s} and Zr_{3d} photoelectrons remain still unclear. However, the chemical shifts of O_{1s} and Zr_{3d} photoelectrons decreased after

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Table 1 Values of O_{1s}, $Zr_{3d(5/2)}$ and $Fe_{2p(3/2)}$ binding energies obtained from zirconia surface after reacted with carbon steel.

Sputtering time	O_{1s}	$Zr_{3d(5/2)}$	$Fe_{2p(3/2)}$
5 min	535.0 eV	187.0 eV	710.4 eV
30 min	533.4 eV	185.4 eV	no detection

30 min Ar^+ ion sputtering and Fe_{2p} peaks disappeared. Therefore, the anomalous chemical shift of O_{1s} and Zr_{3d} photoelectrons may be closely related dissolution of ferric ions into zirconia. Further, it is suggested that the reaction layer of ferric ions is about 300Å at most because Ar^+ ion sputtering rate in this experiment was about 10 Å/min.

In further investigation, factor analysis for enhanced reaction of iron to zirconia should be desired.

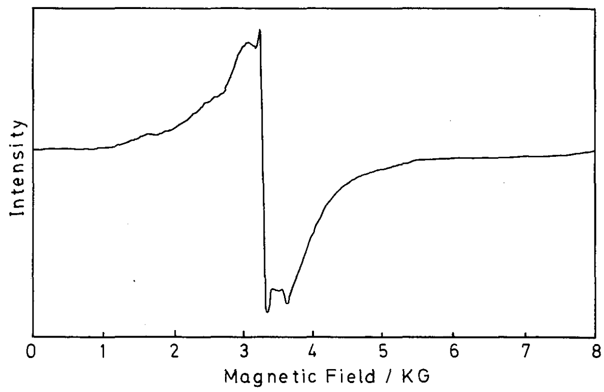

Fig. 3 ESR spectrum obtained from zirconia reacted with carbon steel.

References

1) Handbook of X-ray photoelectron spectroscopy, Published by Perkin-Elmer corporation (1979).

2) for example, N. Iwamoto, Y. Makino and S. Kasahara J. Non-Crystalline Solids, 55 (1983) p 113.