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Investigation of Al_2O_3/Cr_3C_2 composites prepared by pressureless sintering: 3

Jow-Lay Huang[a,*], Jin-Jay Huang[a], Ching-An Jeng[a], Ai-Kang Li[b]

[a]*Department of Materials Science and Engineering, National Cheng-Kung University, Tainan, Taiwan 701*
[b]*Materials Research Laboratory, Industrial Technology Research Institute, Hsinchu, Taiwan 310*

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Abstract

The phase stability of Cr_3C_2 in Al_2O_3/Cr_3C_2 composites sintered in as received Ar, purified Ar and vacuum were investigated. Test samples were cold isostatically pressed prior to pressureless sintering. Results suggested that phase conversion of Cr_3C_2 was eliminated by the reduction of oxygen content in sintering atmosphere. In addition, the porosities were substantially reduced, and strength and toughness were enhanced while sintered in vacuum. © 1999 Elsevier Science Limited and Techna S.r.l. All rights reserved

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1. Introduction

Chromium carbide has been successfully incorporated into alumina for toughening purpose, owing to its high Young's modulus and high temperature erosion resistance. Promising mechanical properties along with high temperature oxidation resistance of Al_2O_3/Cr_3C_2 have been previously reported [1–4]. The phase stability, crack behaviour, microstructure and mechanical properties of pressureless sintered Al_2O_3/Cr_3C_2 composites were previously investigated. The oxygen content had substantial effects on the chemical stability of chromium carbide as was proposed [5,6].The effects of sintering atmosphere on the phase stability, porosities, strength and toughness of Al_2O_3/Cr_3C_2 are reported here.

2. Experimental Procedure

Alumina powder (A16-SG, Alcoa, USA, 0.5 μm) was ball milled with Cr_3C_2 (grade 160, H.C. Starck, Germany, 2 μm) in a polyurethane bottle with high alumina balls and ethanol for 24 h. The ratio of ball to charge to vehicle was 6:1:5 by mass. The quantity of chromium carbide in the mix ranged from 10 to 40% by volume. The slurry was then dried in a rotating vacuum condenser. Dried agglomerates were ground with alumina mortar and pestle, and screened through a 100 mesh sieve for pulverising aggregates.

The powder mixtures were die-pressed to form bars, and cold isostatically pressed under a pressure of 100 Mpa for 1 min. Samples were then loaded in a graphite crucible in powder bed, and sintered at different temperatures for 2 h in a graphite furnace (Centorr/Vacuum Industries, Inc., model 10-2). The packing powders composed of 50 wt% BN and 50 wt% powders of the same composition as the sintering samples. The graphite provided a relatively reducing environment. The atmospheres used in this study include as received Ar (99.99 pure), purified Ar (purified by passing through Copper at 600°C) and vacuum (290 Nm^{-2}).

Density was measured by the water displacement technique. Flexural strength was determined by four-point bending on a universal testing instrument (Shimadzu AGS-500) at a displacement of 0.5 mm min^{-1}. The outer and inner spans were 30 mm and 10 mm, respectively. The nominal dimensions of the testing bars were 3×4×40 mm with 45° edge chamfers. Fracture toughness was measured by a single edge notched beam (SENB) technique. The precracks were cut by a 0.15 mm diamond blade to a depth of 1.33 mm. Each data point represents an average of seven tests.

* Corresponding author. Tel.: +886-6-234-8188; Fax: +886-6-234-6290.

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Phases were analysed by X-ray diffractometer (Rigaku D/max IIB, Japan) using a Cu target and Ni filter. The scanning rate was 0.5° min^{-1} from 32 to 56° or 4° min^{-1} from 20 to 80°.

3. Results and Discussion

The X-ray diffraction results of Al_2O_3/Cr_3C2 composites sintered at 1450 and 1550°C 2 h in as received and purified Ar are shown in Fig. 1. The content of Cr_7C_3 phase evidently increased with the increase of temperature. No trace of phase conversion was observed in samples sintered in purified Ar (Fig. 1c) in which the oxygen was substantially reduced by passing over hot Cu at 600°C. Results suggested the reduction of oxygen in atmosphere could effectively inhibit the phase conversion of Cr_3C_2. Purified Ar was then used for the following experiments in this study.

Powder mixtures and compacted bars of Cr_3C_2/Al_2O_3 were sintered at 1550°C, 2 h, under vacuum and analysed by X-ray diffractometer with a slow scanning rate of 0.5° min^{-1} from 32 to 56° (Fig. 2). No trace of Cr_7C_3 was detected.

The relative density of Cr_3C_2/Al_2O_3 composites as functions of Cr_3C_2 content and sintering temperature are shown in Figs. 3 and 4 respectively. Since no Cr_7C_3 was detected, the relative density was calculated by defining theoretical density as a mixture of solid phases, i.e. Cr_3C_2 and Al_2O_3. Samples sintered under vacuum invariably had greater density than samples sintered in Ar. The trapped Ar gas was probably responsible for its relatively low density [7]. A maximum density of over 98% TD, slightly higher than that of monolithic alumina, was obtained for 10 vol% Cr_3C_2/Al_2O_3 .This was probably due to the inhibition of grain boundary migration by Cr_3C_2 [8]. The densification decreased however, with the increase of Cr_3C_2. The pore size distribution of 40 vol% Cr_3C_2/Al_2O_3 samples sintered at 1550°C 2 h were measured by mercury porosimetry (Micrometrics Instrument Co., Autopore II 9215, USA) and represented in Fig. 5. Results revealed that lower porosities occurred in samples sintered in vacuum.

The strength and toughness of monolithic alumina and Cr_3C_2/Al_2O_3 composites are shown in Figs. 6 and 7. The strength and toughness of alumina were substantially enhanced by the addition of Cr_3C_2 although degradation occurred in samples containing 40 vol%

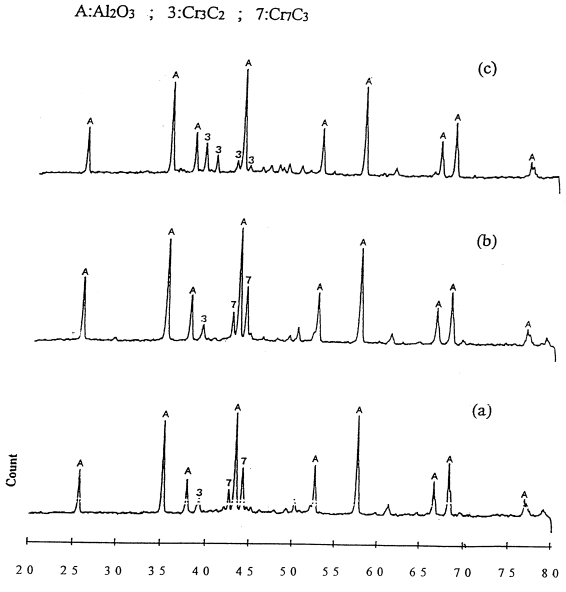

Fig. 1. X-ray diffraction patterns of Cr_3C_2/Al_2O_3 sintered at (a) 1450°C, in as-received Ar; (b) 1550°C, in as-received Ar; (c) 1550°C, in purified Ar.

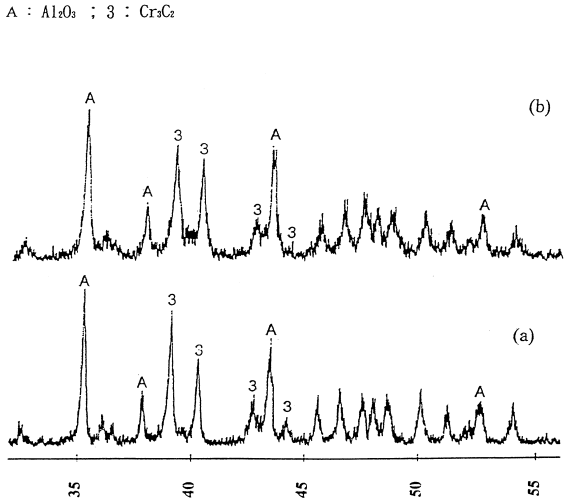

Fig. 2. X-ray diffraction patterns of Cr_3C_2/Al_2O_3: (a) powders, (b) bulks. Samples were sintered at 1550°C, 2 h under vacuum.

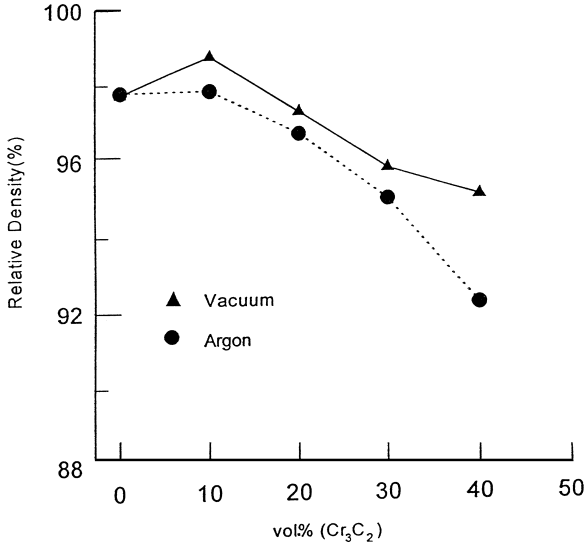

Fig. 3. Relative density vs Cr_3C_2 content.

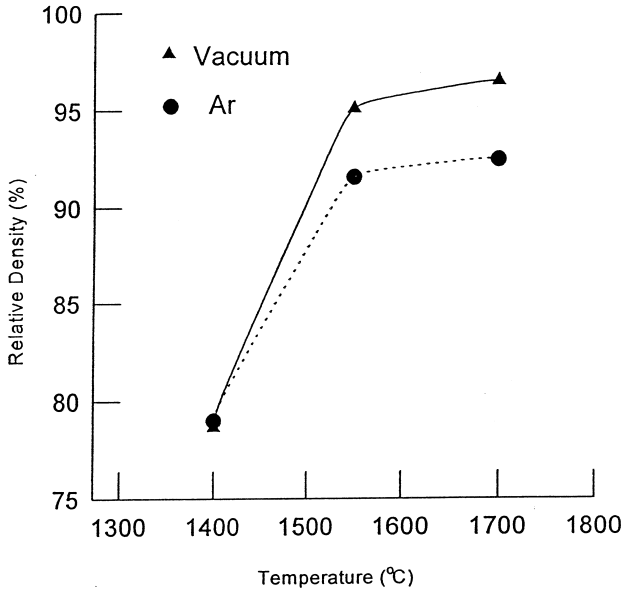

Fig. 4. Relative densities of 40 vol% Cr_3C_2/Al_2O_3 sintered at different temperatures.

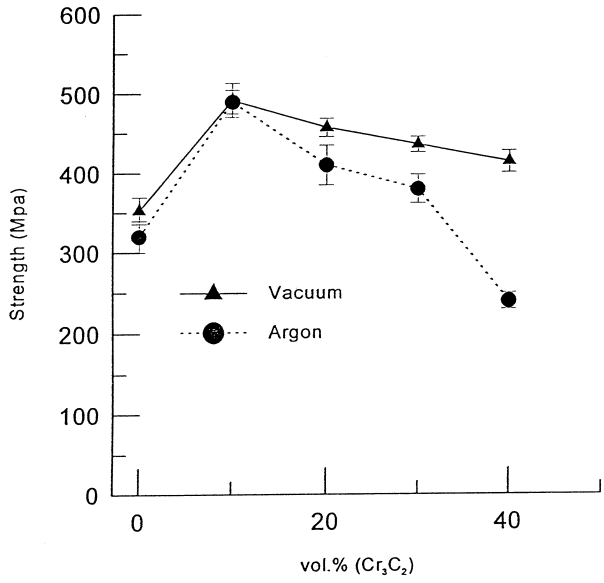

Fig. 6. Strength vs Cr_3C_2 content in Cr_3C_2/Al_2O_3 composites. Samples were sintered at 1550°C, 2 h.

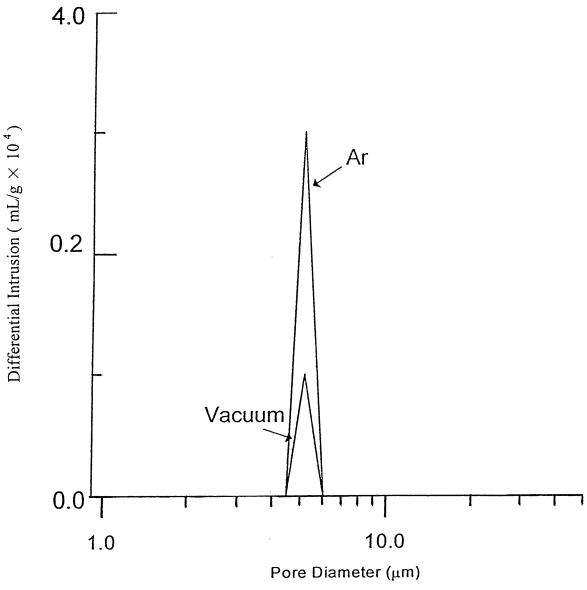

Fig. 5. Distribution of porosities in 40 vol% Cr_3C_2/Al_2O_3 composites sintered at 1550°C, 2 h.

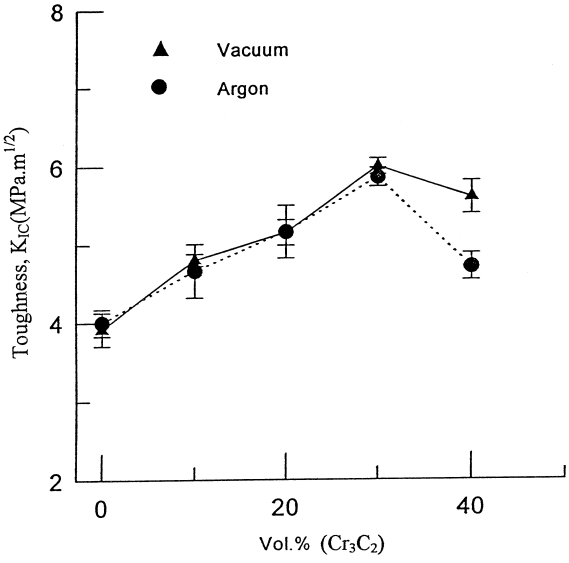

Fig. 7. Toughness vs Cr_3C_2 content in Cr_3C_2/Al_2O_3 composites. Samples were sintered at 1550°C, 2 h.

Cr_3C_2. Both strength and toughness of samples sintered in vacuum were consistently greater than when sintered in Ar.

Microcracks were introduced on the finely polished surfaces of samples by a Vickers diamond indentor (Akashi AVK-A) at 50 Kg for 15 s. Examination revealed that the propagating cracks were inhibited by Cr_3C_2 particles. This effect was especially pronounced in composites sintered in vacuum than in Ar, despite their relatively less porosities.

4. Summary and Conclusions

1. Phase stability of Cr_3C_2 at elevated temperatures in as received Ar, purified Ar and vacuum were compared. Results suggested that phase conversion of Cr_3C_2 was eliminated by the reduction of oxygen content in sintering atmosphere.

2. Porosities were reduced, density, strength and toughness were enhanced in samples sintered in vacuum than in Ar.

Acknowledgement

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