

PREPARATION OF Al_2O_3 CERAMIC MATRIX IN - SITU COMPOSITES BY REACTIVE MELT PENETRATION

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Abstract

Reactive metal penetration is a novel method for the preparation of $\text{Al}_2\text{O}_3/\text{Al}$ matrix composites. In metal matrix in-situ composites generally the reinforcing phase is produced by chemical reaction in the molten matrix phase. However, in the reactive metal infiltration process the matrix phase, viz. Al_2O_3 is produced in-situ by chemical reaction between SiO_2 and molten aluminium,



The paper deals with the preparation of the in-situ composite and its microstructural features. The method offers the possibility of producing net-shape or near net-shape composites.

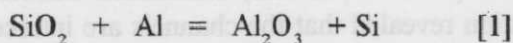
Introduction

Aluminium oxide has several remarkable properties for use as a cutting material. However, its shortcomings are low values of tensile strength and toughness. Transverse rupture strength and toughness are influenced by density and microstructure, i.e. shape, size and distribution of the grains. It has been found that addition of TiC , TiN or SiC , (10 - 20%) will improve the toughness considerably. Addition of silver metal has been found to inhibit grain growth during sintering and improve toughness [1]. In-situ process involving chemical reactions to produce one of the phases of the composite offer several benefits like thermodynamic compatibility of the phases, uncontaminated interfaces and control of particle size. This enables uniform distribution of the phases and eliminates segregation.

Recently $\text{Al}_2\text{O}_3/\text{Al}$ composites were produced by the direct metal oxidation (DIMOX) process. This involves oxidation of molten aluminium at high temperature ($T > 900^\circ\text{C}$) [2]. At such high temperature a layer of oxide (Al_2O_3) grow continuously on the surface of the molten metal and channels of molten aluminium are embedded in it. The composition and growth of the oxide layer are dependent on alloy composition and temperature.

Another new and promising process for in-situ synthesis of $\text{Al}_2\text{O}_3/\text{Al}$ compos-

ites involves the use of the well known thermit reaction between molten Al and an oxide [3-9]. On account of the very high stability of aluminium oxide most of the other oxides are reduced to the respective elements by aluminium when heated to the appropriate temperature. Reaction synthesis of Al₂O₃/Al composite using SiO₂ and aluminium was reported by Matsuo and Inaba [3], who reacted amorphous silica with molten aluminium at above 900°C. Preparation of similar composites using amorphous silica and Al were also reported by Breslin et al., in 1994 [4]. The controlled reaction between silica and aluminium resulted in the formation of mutually interpenetrating composite structure of Al₂O₃ and Al (Si)



The aluminium content of the composite can go up to about 30%. On account of the high fracture toughness and thermal conductivity of Al the composite also exhibited significant improvement in these properties. Recently Loehman et al. [7-9] showed that Al₂O₃/Al composites can also be prepared by silicate ceramic preforms. They also synthesized these composites making use of hot pressed mixtures of powders of mullite and aluminium. When the preform is immersed in molten aluminium, silica reacts with the metal to form Al₂O₃ and Si, and channels of Al are embedded in the alumina. If an excess of aluminium is present in the molten bath then the silicon produced in the chemical reaction diffuses out into the reservoir and the composite consists of almost pure Al and Al₂O₃ [6,8]. On the other hand when hot pressed powders are used the composite consists of Al₂O₃/Al/Si, i.e. Si produced in the chemical reaction is retained in the composite. Gao et al. [6] have shown that in composites produced by the reactive metal penetration process the Al channels contain about 1 wt% Si. The use of powder metallurgical route or alumino silicate ceramics enable the variation of the volume fraction of the Al phase in the composites by careful control of the Al₂O₃ : SiO₂ ratio. Some properties of the ceramic matrix composites (CMC) as a function of Al(Si) content is shown in Table 1 [8]. Since the size and shape of the composite produced by the reactive melt penetration process are related to those of the preform, the process enables the preparation of net-shape products of the composites. The present paper deals with some preliminary results of the preparation of net-shape in-situ Al₂O₃/Al composites by the reactive melt penetration process.

Experimental Procedure

For the experimental studies amorphous silica and dense sillimanite tubes were used. Commercial grade aluminium metal was melted in a graphite crucible in a resistance furnace maintained at 900-1000°C. The preforms in the shape of cylindrical specimens of 10mm diameter and 20mm length and tubes of 12mm outer diameter and 25mm in length cut from commercial material were used. When molten metal was at the required temperature, the samples were carefully immersed into the molten bath

with the help of a graphite rod. Periodically the samples were taken out to determine the extent of reaction. Aluminium adhering to the samples was removed with the help of a knife. The specimens were polished in the conventional manner and examined in the optical microscope.

Results

The samples of the composites were dark grey in colour. Microscopic examination of the samples revealed the presence of aluminium in the form of an uniform distribution of fine ($\sim 0.2\text{--}0.5\mu\text{m}$ dia.) aluminium channels, as shown in Figure 1. Further examination revealed that the channels are interconnected. The microstructure is very similar to that observed in composites made by direct metal oxidation (DIMOX) process. In the DIMOX process addition of Mg or Zn to the aluminium bath was reported to facilitate the formation of the composite. However, the use of dilute (1-2%) Al-Mg/Zn alloys has not been found to be of any help in the experiments. This observation is same as that reported by Loehman et al. [6-8], who found the use of aluminium of the highest purity to give best results. Loehman et al. carried out the experiments in purified argon atmosphere. But our results show that composite synthesis can be carried out even in ambient atmosphere.

Conclusions

1. $\text{Al}_2\text{O}_3/\text{Al}$ Ceramic matrix composites can be prepared from dense aluminosilicate preforms by reactive metal penetration process.
2. Composite synthesis can be carried out in air.
3. Commercial grade aluminium can be used for the preparation of in-situ $\text{Al}_2\text{O}_3/\text{Al}$ (Si) composites.
4. As the shape and size of the composite are related to those of the preform, net-shape products can be produced.

References

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Table 1
Properties of in-situ composites as a function of Al (Si) content

Property	Al(Si) 0	Al(Si) 4.7	Al(Si) 15	Al(Si) 22	Al(Si) 26
Vol % Al					
Density, g/cc	3.73	3.65	3.72	3.46	3.46
E, GPa	354	324	294	281	277
G, GPa	144	131	117	114	111
Poisson Ratio	0.23	0.23	0.25	0.24	0.27
Coef. of ther. expan. RT - 300°C					
µm/m/°C	6.8	7.1	8.7	7.6	8.6
VPN, GPa	15.7	13.8	13.6	8.8	8.5
Toughness, MPa.m ^{1/2}	3.9	4.5	—	5.5	6.5
Bend strength MPa	302	278	—	280	—

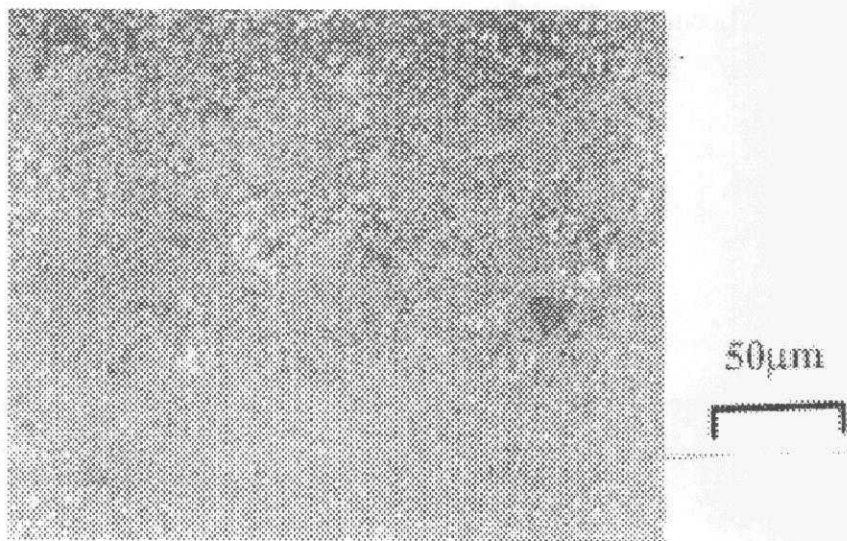


Fig. 1 - Microstructure of in-situ $\text{Al}_2\text{O}_3/\text{Al}$ composite showing the fine distribution of Al channells (400x)