STUDIES ON THE IN-SITU FORMATION OF Al_2O_3 - SiC CERAMIC MATRIX COMPOSITE FROM TATA STEEL COLLIERY OVER BURDEN

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Abstract

Raw materials suitable for the production of in-situ ceramic matrix composites (CMC) are scarce. In the present investigation, colliery over burden has been identified as a promising feedstock for the preparation of Al_2O_3-SiC CMC. Colliery minestones from Tata Steel colliery has been assessed chemically and found to contain alumina, silica and carbon. After preliminary processing of minestones, high temperature synthesis has been carried out in the laboratory-scale as well as relatively larger scale to produce CMC.

The results of the investigation have shown that substantial amount of silicon carbide whiskers and particulates could be produced in the alumina matrix. Bench scale as well as larger scale production trials have been found to be successful in terms of yield of SiC and its quality.

Additionally, in-situ Al_2O_3-SiC CMC has been found to contain carbon over and above required for the formation of SiC. Thus Al_2O_3-SiC-C CMC has been gainfully utilised in the preparation of aluminium based metal matrix composite with distinct advantages of higher strength combined with improved ductility.

Introduction

In the continuing quest for improved performance which may be specified by various criteria including less weight, more strength and lower cost, currently used materials frequently reach the limit of their usefulness. Thus material scientists and engineers are always striving to produce either traditional materials or completely new materials. Composites are examples of the latter category.

A composite is a material having two or more distinct phases on a microscopic scale separated by a distinct interface. The constituent that is continuous and is often present in greater quantities in the composite is termed as matrix. The properties of the matrix are improved on incorporating another constituent known as the reinforcing phase as it enhances the mechanical properties of the matrix. In most of the cases the reinforcement is harder, stronger and stiffer than the matrix with the exception of ductile metal reinforcement in a ceramic matrix.

Conventionally Al based metal matrix composite is prepared by the addition of SiC particulate or whiskers, Al_2O_3, SiO_2, graphite etc., singly or in combination i.e. by mechanical mixing. The present investigation aims at producing Al based metal matrix composite by reinforcing the melt with in-situ formed Al_2O_3 - SiC from colliery over burden. As a first step, the in-situ CMC is prepared by high temperature treatment of colliery over burden which basically contains alumina, silica and carbon. The liquid metallurgy route has been adopted for the production of MMC. The reinforcing agent Al_2O_3 -SiC in-situ composite contains carbon in addition to alumina and silicon carbide. The presence of carbon is beneficial in improving elongation of the Al based MMC compared to conventional MMC without carbon.

2.0 Experimental methods and materials

2.1 Preparation of Ceramic Matrix Composite

The colliery minestone collected from Tata Steel colliery was ground and sieved below 44 micron. 15 gm of the sample was taken in graphite crucible and high temperature reactions were conducted in platinum wound resistance furnace at various temperatures for different length of time and the optimum conditions were determined. Using the optimised results upscaling of the preparation of CMC was done at 1600°C for two hours in Dynatech furnace.

The ground and sieved powder was mixed with polyvinyl alcohol as binder. 50mm x 20mm thick cake of the above sample was made in a hydraulic press in metallic die. The pressure applied was 5-7 kg/cm^2. The prepared cake was taken into a graphite crucible of about 1 kg capacity. This was heated in a graphite resistance furnace at 1600°C for 2 hours in the atmosphere of argon. The material was cooled in the furnace to room temperature maintaining argon atmosphere.

2.2. Production of Al based Metal Matrix Composite

2.2.1. Preparation of the Aluminium Melt.

An electric furnace was employed for melt preparation. Weighed quantity of aluminium was taken in a graphite crucible and placed at the bottom of the electric furnace. A temperature of 750-800°C was maintained for melting 500 g - 1 kg Aluminium. The CMC was taken in a crucible and preheated to 750°C in an electrically heated furnace. Different percentage addition of CMC was made to the melt followed by mechanical stirring device. The stirring continued for 2-4 minutes depending upon the melt quantity and amount of CMC added. The amount of CMC addition varied from 5 - 20% by weight.

In another experiment an induction furnace of 10 kg capacity was used to melt aluminium and electromagnetic stirring device was used for particulate reinforcement.

2.2.2. Casting of aluminium based MMC.

After thorough stirring of the melt it was poured in ingot moulds to cast 150 × 6 mm specimen.

2.2.3. Specimen preparation for characterization

Specimens were prepared for conducting mechanical tests such as ultimate tensile strength and hardness. The dimensions of the samples were as follows :

Diameter = 6 mm
Parallel length = 30 mm

For hardness determination cylindrical samples were used and for structural evaluation SEM was used. The cast samples were examined with regard to the phases present using XRD.

3.0 Results and Discussions

3.1 Preparation of CMC from colliery minestones

Colliery minestones were collected from two different sources namely,.

(i) West Bokaro — Tata Steel Colliery

(ii) Western Coal Field Ltd. — Nagpur

The materials were analysed and found to be chemically consistent. Table I gives the chemical compositions of the minestone.

Table I
Chemical Analysis of Colliery over burden

Chemical Constituents	Percentage Analysis Sample I	Sample II
Al_2O_3	32	33.5
SiO_2	42	40.8
C	25	23.7
Fe_2O_3	0.5	1.5

Sample I : Colliery over burden from West Bokaro.
Sample II : Colliery over burden from WCL Nagpur.

The samples contained appropriate Al_2O_3 and SiO_2 ratio and amount of carbon for the preparation of in-situ CMC. Further, additional carbon left behind may be gainfully utilized in the preparation of Al based MMC. Carbon is known to enhance the ductility of the MMC.

The possible reaction path in-situ may be represented chemically as

$$SiO_2 + 3C = SiC(s) + 2CO(g)$$
$$2Al_2O_3 + 3C = Al_4O_4C(s) + 2CO(g)$$
$$Al_6Si_2O_{13}(s) + 6C = 3Al_2O_3(s) + 2SiC(s) + 4\ CO(g)$$
$$SiO_2\ (s) + CO(g) = SiO\ (g) + CO_2(g)$$
$$SiO(g) + 2C = SiC_{(s)} + CO(g)$$

At higher temperature around 1400°C alumino-silicate present in the minestone becomes very unstable and the crystal lattice disrupts favouring the thermo-chemical in-situ reaction with carbon.

3.2. Characterisation of CMC

From the XRD analysis (Fig.1) it is interesting to observe that the reaction has produced beta-silicon carbide (silicon carbide whiskers) and alpha alumina. This further suggests that the carbon present in the colliery over burden is in the amorphous form since amorphous form of carbon can only produce whiskers in the high temperature synthesis.

SEM studies (Fig.2) has revealed adequate formation of β-silicon carbide in the matrix of alumina. From stoichiometric calculation it has been observed that some loss of silica has taken place and this loss may be attributed to the formation of SiO vapour. The loss of silica has been around 5%. Loss of carbon is around 3%.

3.3 Preparation of aluminium based MMC.

The preparation of aluminium based MMC was done using addition of alumina and silicon carbide singly and in combination. The results of 15 and 20% additions have been reported in this paper. The in-situ composite containing Al_2O_3 and SiC was added to one set of experiment and to another Al_2O_3-SiC-C has been added.

3.4. Characterisation of the Al based MMC.

XRD analysis has confirmed (Fig.3) the presence of the phases namely Aluminium, alumina and silicon carbide.

The ultimate tensile strength of the MMC containing alumina and silicon carbide have been presented in table II and table III respectively.

Table II
Effect of Alumina addition on the UTS

Reinforcment with Alumina	UTS (MPa)
5%	105.3
10%	180.9
15%	250.0

Table III
Effect of SiC addition on the UTS of MMC

Reinforcement with SiC	UTS (MPa)
5%	260.5
10%	280.7
15%	310.0

It is observed from the tables that silicon carbide particulate addition singly registers higher strength compared to similar addition of alumina. Table IV and Table V show the UTS and elongation of MMC with the addition of Al_2O_3, SiC and carbon in combination.

Table IV

Effect of addition of Al_2O_3-SiC In-Situ CMC on the Mechanical Properties*.

% Composition	UTS (MPa)	Hardness (VPN)	% Elongation
Al + 6% Al_2O_3 + 5% SiC	281.0	170	Nil
Al + 15% Al_2O_3 + 10% SiC	312.5	200	Nil
Al + 25% Al_2O_3 + 20% SiC	340	250	Nil

** Carbon was burnt off from the in-situ CMC before addition to the Al melt.*

Table V
Effect of addition of Al_2O_3-SiC-C in-situ CMC on the Mechanical Properties of the MMC

% Addition	UTS (MPa)	Hardness	% Elongation
Al + 6% Al_2O_3 + 5% SiC	280.0	160	3.0
Al + 15% Al_2O_3 + 10% SiC + 5% C	315.0	180	4.4
Al + 20% Al_2O_3 + 20% SiC + 8% C	338	200	5.2

An overall improvement in mechanical properties of Al based MMC has been observed with the addition of in-situ Al_2O_3-SiC-C CMC prepared from colliery over burden.

The improvement in the mechanical properties may be attributed to homogeneous mixing of Al_2O_3-SiC-C CMC resulting in uniform distribution. The phases namely Al_2O_3 and SiC have nucleated in combination during the high temperature synthesis. This has led to overall improvement in the mechanical properties.

5. Conclusions

- Colliery over burden has suitable chemical composition in respect of alumina, silica and carbon necessary for the formation of in-situ Al_2O_3-SiC-C CMC.
- Silicon carbide whiskers can be successfully produced by using high temperature synthesis with distinct economic advantage from abundantly available indigeneous sources.
- In view of ease of preparation of in-situ Al_2O_3-SiC-CMC, handling of individual particles of SiC can be eliminated and thus health hazards can be reduced significantly.

- Presence of excess carbon in the CMC has been gainfully utilised in the preparation of Al based MMC.
- In addition to high strength of Al based MMC the ductility of the product has been found to improve compared to conventional addition of CMC.
- XRD studies of CMC have shown Al_2O_3-SiC phases more predominantly.
- Electromagnetic stirring for reinforcement has been found superior to mechanical stirring.

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Selected Bibliographies

1. P.K.Rohatgi, "Metal Matrix Composite", Defence Science Journal, **43** (4) (1973) 323-349.
2. H. Lagale and D.J.Loyd, Microstructural Analysis of Al-SiC composites, Canadian Metallurgical Quarterly, **2** (2) (1989) 145-152.
3. M.R. Perugonda, A.C.D. Chaklader, Alumina - SiC composites from kaolinite-carbon precursors by hot pressing, Solid state phenomena, **839** (1989) 457-470.
4. S.F. Moustafa "Technical Note" Canaddian, Metallurgical Quarterly, **33** (3) (1994) 259-264.
5. Y.B. Liu, S.C.Lim, M.O. Lai , Recent development in the fabrication of metal matrix particulate composites using powder metallurgy technique, J. Mater. Sci., **29** (1994) 1999-2007.
6. P.M. Mummery, B.Derby, J. Mater. Sci., **29** (1994) 5615-5624.
7. Z.F. Zhang, L.C. Zhang, Particle effects of friction and wear of aluminium matrix composites, J. Mater. Sci., **30** (1995) 5995-6004.

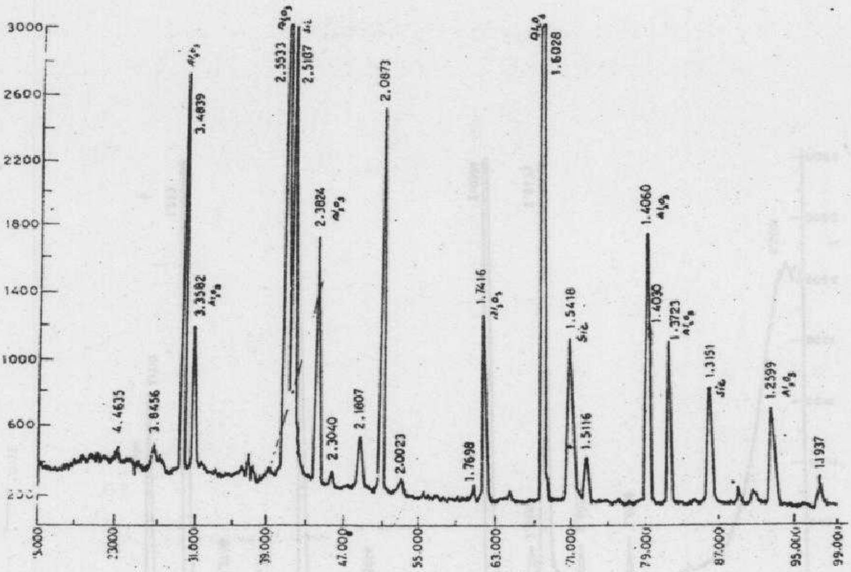

Fig. 1 - XRD Pattern of Alumina-Silicon Carbide composite.

Fig. 2 - In-situ formation of Al_2O_3-SiC X1000

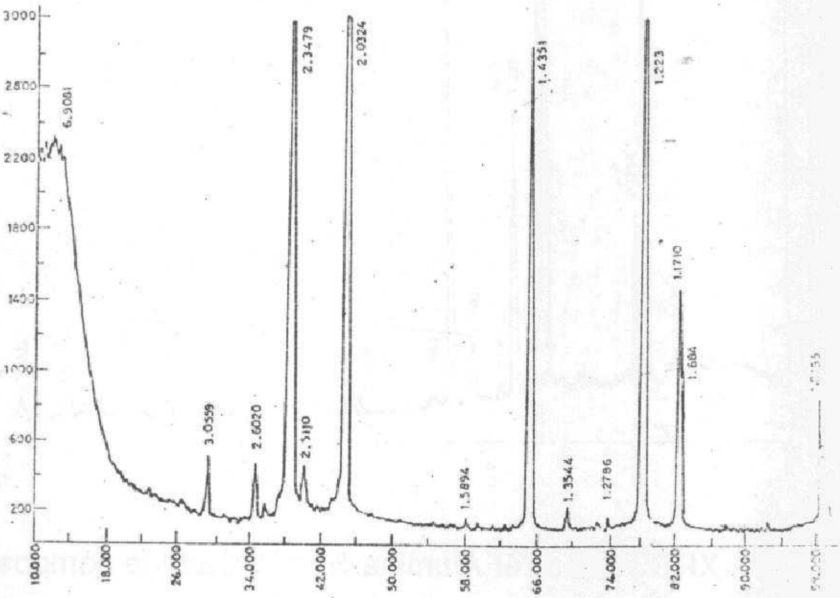

Fig. 3 - XRD Analysis of phases Al, Al_2O_3, SiC in Al based MMC