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Decomposition reactions in the SiC–Al–Y–O system during gas pressure sintering

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Abstract

The substantial densification, that occurred in the SiC–Al–Y–O system was explained in the present work by analysing possible chemical reactions and their dependence on initial particle associations, i.e. homogeneity of mixing, the physical and chemical state of additives, pressurised sintering environment over the reactants and temperature of sintering. Hydroxyhydrogel powder precursors were found to be better than mechanically mixed SiC–YAG powder and pre-forming of YAG by holding the specimens at the temperature of 1400°C for 2 h were found to be the best. Decomposition reactions within the system could be controlled by using finer SiC and applying gas pressure over the reactants. © 2001 Elsevier Science Ltd and Techna S.r.l. All rights reserved.

Keywords: Decomposition reactions; Gas pressure sintering; Hydroxyhydrogel; SiC–YAG composite

1. Introduction

SiC could be sintered by using boron [1–5], aluminium [6] and carbon [7] or by using various metal oxides [8–12] and nitrides [13–16]. Sintering of silicon carbide by B and C additions was suggested [1–5,7] to be controlled by grain boundary diffusion phenomenon. Sintering temperature of such systems was above or around 2000°C. Sintering temperature was much lower when metal oxides [8,17] or nitrides [18] were used as sintering additives. Sintering in such systems was proposed [19–22] to be controlled by liquid phase formation.

Among various metal oxide additives, Al_2O_3–Y_2O_3 combinations have drawn attention due to ease of densification and less rigid process conditions. Al_2O_3–Y_2O_3 additives formed YAG, which was also identified to be the phase responsible for improvement of fracture toughness [19,21,23] of sintered compacts of SiC–YAG composites. Kim and Kim believed [19] that the eutectic liquid, formed [24], remained along grain boundaries and triple points and recrystallised to α-Al_2O_3 and YAG on cooling after sintering. Solution and reprecipitation were suspected to control the mass transport for densification by several authors [19,25–27]. Lack of decomposition of SiC by Al_2O_3 and Y_2O_3 in the temperature range 2027–2127°C was suggested to be the reason behind densification in this system which was aided by liquid [24,27,28,30,31] formation. Ostwald ripening by solution and reprecipitation was identified [25] to control sintering mechanism in SiC–YAG system.

In all the above work, analysis was mostly limited within the range of temperature where liquid phases were formed. In the present investigation, an attempt is made to analyse the chemistry of changes in the SiC–Y_2O_3–Al_2O_3 system leading to densification of powder compacts with ultimate major phases in the specimens, SiC and YAG. Additives used were the mixture of Al_2O_3 and Y_2O_3 of varying composition either in the form of hydrogel or as preformed YAG and their role in the decomposition reaction and densification was studied.

2. Experimental[1]

α-SiC(SiC-98.8 wt.%, Al_2O_3-0.07 wt.%, Fe_2O_3-0.08 wt.%, C-0.15 wt.%, Si-0.5 wt.%, Carborundum

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[1] The preparation part is protected in India by patent application No. 375/DEL/98.

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Universal, India) of the specification as given in Table 1 was used in the present study. Original α-SiC was coarse (sp. surface area 1.09 m^2/g). The coarse α-SiC was ground to a fineness of over 16m^2/g by attrition milling. The milled powder was purified by following the process described elsewhere in detail [32]. Six different additive compositions were taken varying between 5 and 30 wt.% YAG, shown in respective figures in Section 3, where the results were analysed. In the present work, four sets of experiments were conducted by varying process parameters, as illustrated in Table 2.

2.1. Preparation of powder precursors and specimens

A. In one set, powder precursors were prepared through gelation of the suspension of SiC in $Al(NO_3)_3.9H_2O$ and $Y(NO_3)_3.5H_2O$ aqueous solution, procedure of which was described in details in previous works [33,34]. The material so produced was heat treated at 800°C in air.
B. In another set, powder precursors containing Y_2O_3 and Al_2O_3 were prepared through the intermediate hydroxyhydrogel formation by reacting appropriate amount of $Al(NO_3)_3{\cdot}9H_2O$ and $Y(NO_3)_3{\cdot}5H_2O$ in ammonium hydroxide medium to make YAG preform.

The specimens were prepared in the form of pellets (diameter 18 mm, thickness 6 mm) and bars (65×10×8 mm) by uniaxial pressing at 30 MPa followed by isostatic pressing at 250 MPa.

Table 1
Specification of starting SiC powder

Average diameter (μm)		% Coarse in the range (μm)			Average surface area (m^2/g)
Median	Modal	15–25	6–10	1–4	
2.86	3.68	0.60	13.7	4.3	1.09

Table 2
Description of different sets of samples

Set no.	Description	Sintering conditions
I	Hydrogel derived additive + SiC (coarse)	No hold
II	Hydrogel derived additive + SiC (coarse)	Hold at 1400°C
III	Hydrogel derived additive + SiC (fine)	Hold at 1400°C
IV	YAG + SiC (fine)	No hold

2.2. Sintering

Sintering experiments were carried out by using (i) powder precursors prepared in (A) containing SiC and hydroxyhydrogels of yttrium and aluminium, and (ii) mechanically mixed SiC with pre-formed YAG prepared from the powder precursor made in (B). Specimens of category (i) were sintered by (a) holding the specimens at 1400°C for 2 h in order to form YAG in situ before sintering temperature was reached, and (b) the temperature was increased without any hold to the sintering temperature. Heat treatment schedule followed in the experiments is shown in Fig. 1. Argon atmosphere was used during firing with a gas pressure of 6.33 bar.

2.3. Analysis

Weight change was measured gravimetrically and dimensional change was measured by microscope. DTA and TGA (Netzsch model no. STA-409C) were carried out with powder precursors. XRD (with a Philips PW-1730 X-ray chrystallographic unit provided with a proportional counter PW-1050/70 goniometer) analysis was done on Al–Y hydrogel samples heat treated at 800 and 1400°C and of specimens containing SiC–Al–Y–O sintered at 1950°C. IR spectroscopy (Perkin-Elmer model no. 1615 FTIR spectrometer) was done for selected samples. Microstructure of the sintered specimens was analysed by scanning electron microscopy (SEM) (Leo 410i, UK).

3. Results

3.1. DTA and TGA

DTA and TGA were carried out for Al–Y hydrogel (Al_2O_3:Y_2O_3 of molar ratio 5:3) up to 1200°C. An exothermic peak at 903.4°C characterised the formation of

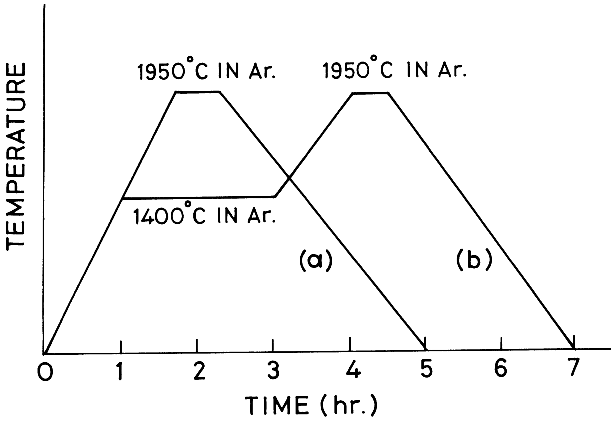

Fig. 1. Firing schedule for different sets of samples: a, sets I and IV; b, sets II and III.

yttrium aluminium garnet [35] (Fig. 2). In presence of SiC (70 wt.% SiC and 30 wt.% YAG), the exothermic peak appeared at 908.4°C. No such peak (~900°C) was found with sample prepared by mechanically mixing yttrium hydrogel and aluminium hydrogel in 5:3 molar ratio (Al_2O_3:Y_2O_3) up to 1200°C (Fig. 3).

3.2. IR studies

The O–H stretching vibration was noted at 3470 cm^{-1} for Al–Y hydrogel system (Fig. 4). Another absorption band was observed at 1383 cm^{-1} which was due to the remaining ammonium salt. The peak for NH_4^+ almost disappeared when IR spectroscopy was done on heat treated (800°C) specimens (Fig. 5). The presence of absorption peak at 3432 cm^{-1} after heat treatment of the specimens at 800°C indicated that even at this temperature hydrogel structure was not completely destroyed [36].

3.3. Weight loss

There was weight loss for all the samples of sets I, II and III. For the samples of set IV, small percentage of weight gain of the specimens was recorded and those were 0.08, 0.17, 0.85, 1.06, 1.58 and 2.06% for 5, 10, 15, 20, 25 and 30 wt.% of YAG-containing specimens, respectively. For set I samples, weight loss increased

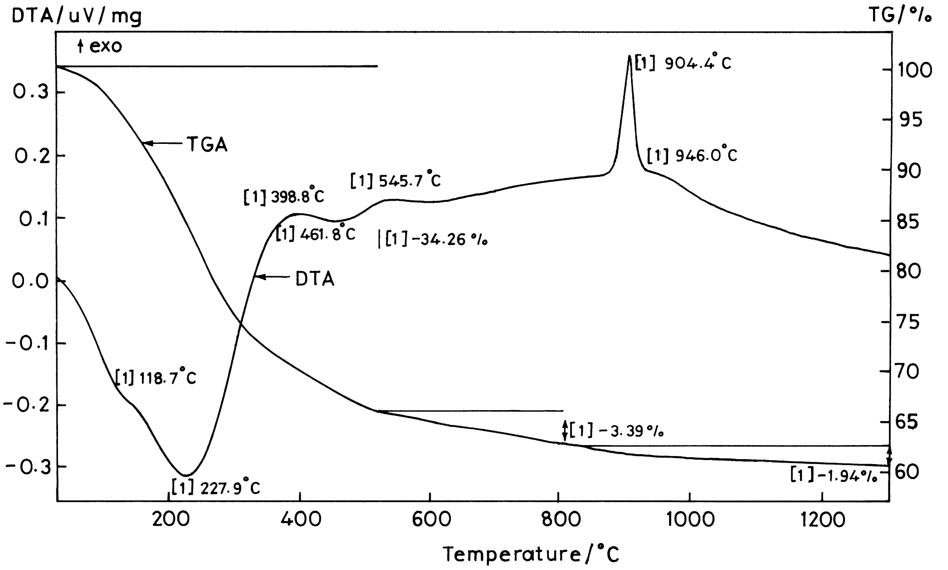

Fig. 2. DTA and TGA of Al–Y hydrogel (Al_2O_3:Y_2O_3 molar ratio 5:3).

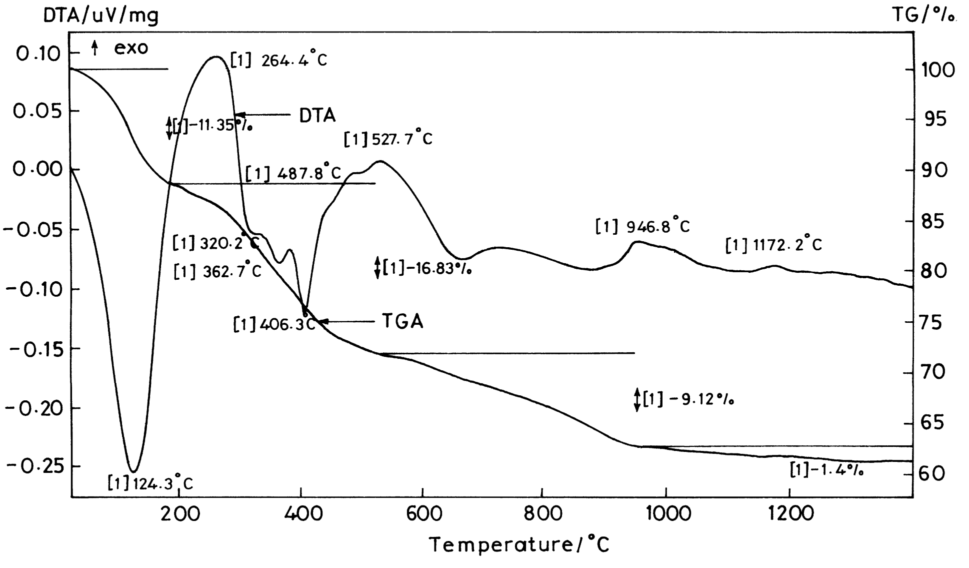

Fig. 3. DTA and TGA of Al_2O_3:Y_2O_3 system (Al_2O_3:Y_2O_3 were derived through hydrogel route separately and mixed mechanically in 5:3 molar ratio).

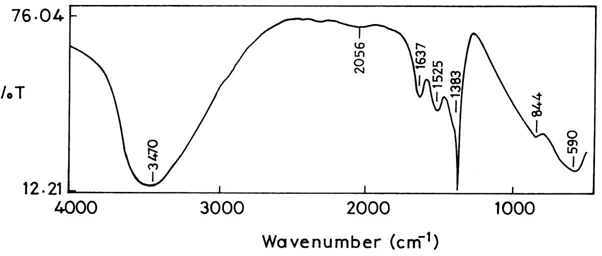

Fig. 4. IR spectroscopy of Al–Y hydrogel (Al_2O_3:Y_2O_3 molar ratio 5:3) heat treated at 110°C.

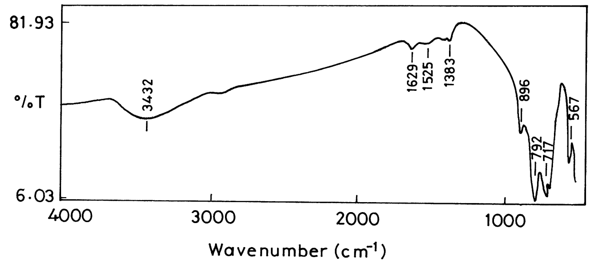

Fig. 5. IR spectroscopy of Al–Y hydrogel (Al_2O_3:Y_2O_3 molar ratio 5:3) heat treated at 800°C.

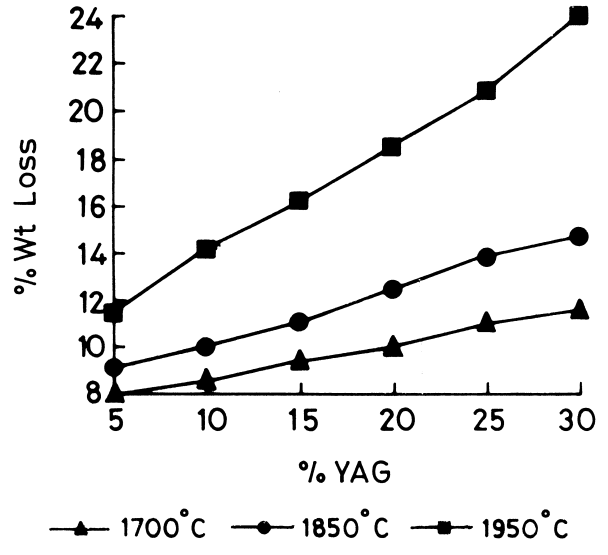

Fig. 6. Weight loss of the specimens in relation to YAG content (set I).

almost linearly with additive (Fig. 6). For set II samples, weight loss increased slowly up to 20 wt.% additive after which it increased steeply (Fig. 7). For set III samples, weight loss decreased continuously with increasing amount of additive (Fig. 8). When samples were heat treated with an intermediate hold at 1400°C, weight loss reduced considerably. The only difference between set I and set III samples was that, in set I, coarse SiC was used but in set III, fine SiC was used. It appeared from Figs. 6 and 8 that weight loss could be reduced by increasing fineness of SiC and the effect was more pronounced with higher amount of additives.

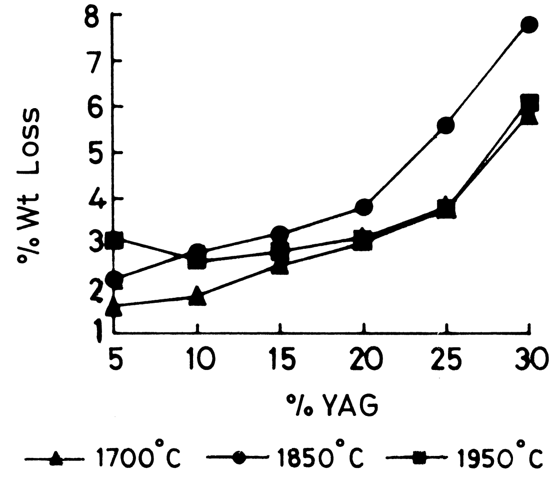

Fig. 7. Weight loss of the specimens in relation to YAG content (set II).

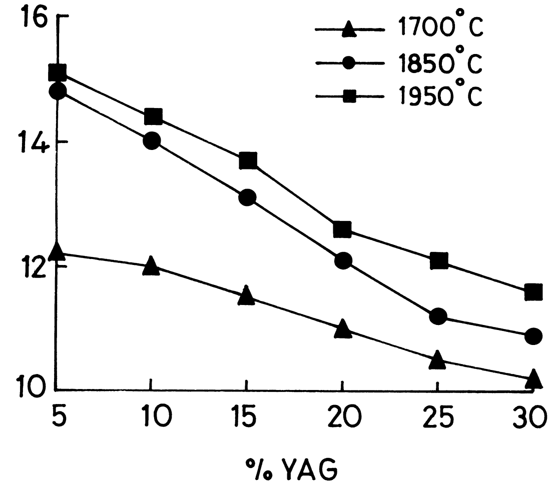

Fig. 8. Weight loss of the specimens in relation to YAG content (set III).

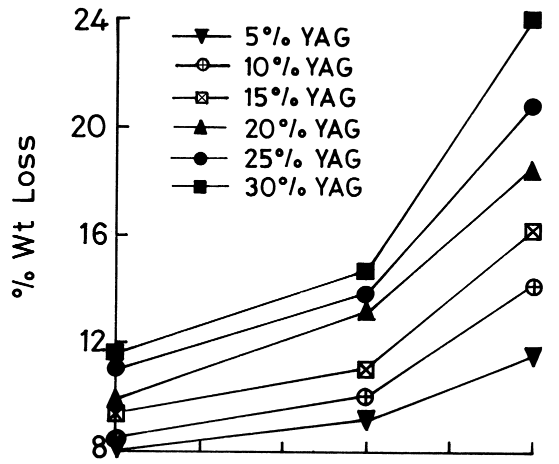

Fig. 9. Weight loss of the specimens with sintering temperature (set I).

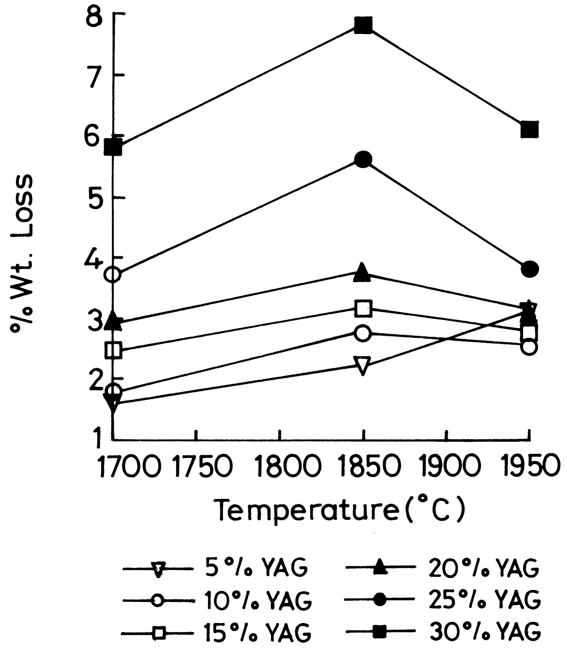

Fig. 10. Weight loss of the specimens with sintering temperature (set II).

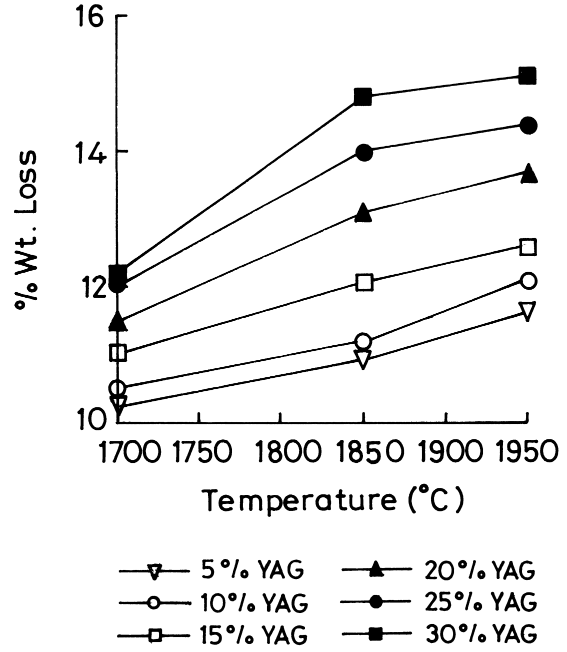

Fig. 11. Weight loss of the specimens with sintering temperature (set III).

Again, weight loss was found to increase slowly up to 1850°C for all the samples in sets I–III (Figs. 9–11). For the samples of set I, there was a steep rise of weight loss from 1850 to 1950°C. In the same temperature range, weight loss remained almost the same for samples of sets II and III. Therefore, the 'hold' of the samples at 1400°C reduced weight loss to a considerable extent.

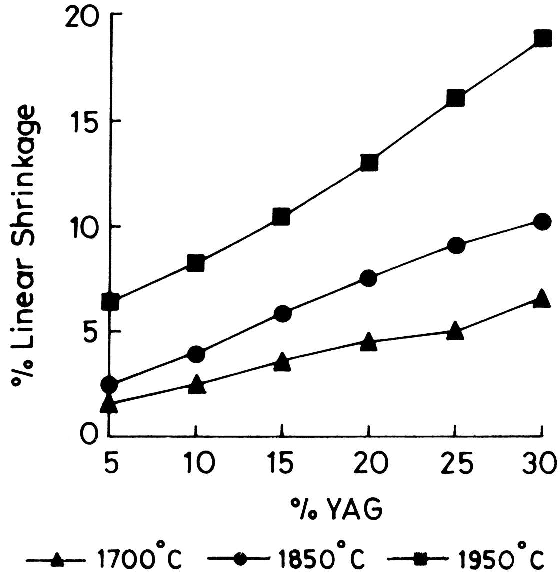

Fig. 12. Linear shrinkage of the specimens in relation to YAG content (set I).

3.4. Linear dimensional change

Extent of sintering was expressed as percent linear shrinkage and is represented in Figs. 12–19. In all the systems, sets I–IV, shrinkage increased with increasing additive concentration (Figs. 12–15). Maximum shrinkage was observed for sets I and III (Figs. 12 and 14). Substantial amount of shrinkage was also observed for set II where an intermediate hold at 1400°C was maintained (Fig. 13). Shrinkage was minimum in set IV where pre-formed YAG was mixed with SiC mechanically. Linear shrinkage was found to increase rapidly from 1850°C for all the specimens in sets I–III. Therefore, it may be concluded here that a mere mechanical mixing of SiC and YAG did not produce good sintered materials. In-situ formation of YAG in the reaction system was always beneficial for obtaining better densification. It may also be noted that though comparable shrinkage of the samples was obtained with or without hold at an intermediate temperature, the former was a better method for producing sintered compacts because of lower weight loss if we consider the weight loss parameter along with shrinkage.

3.5. XRD analysis

XRD of Al–Y hydrogel heat treated at 1400°C indicated complete conversion of Al_2O_3 and Y_2O_3 to YAG (Fig. 20). When SiC was present in hydrogel precursors, small amount of unreacted Al_2O_3 and Y_2O_3 was detected in specimens containing $\geqslant$80 wt.% SiC after heat treatment at 1400°C for 2 h [33]. This was due to dilution of Al_2O_3 and Y_2O_3 in the system by SiC resulting into retardation of reaction between Al- and

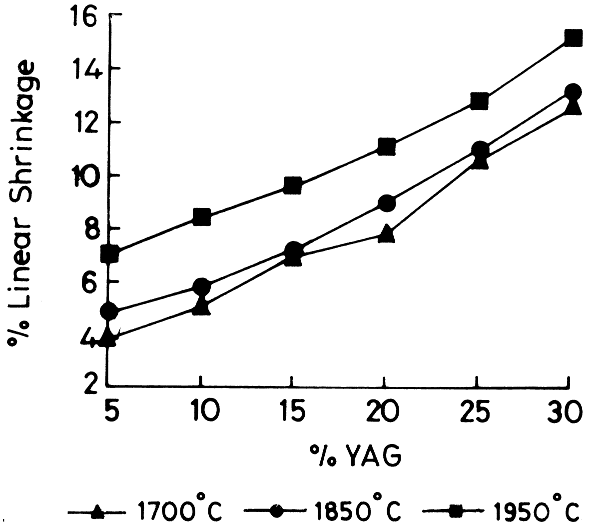

Fig. 13. Linear shrinkage of the specimens in relation to YAG content (set II).

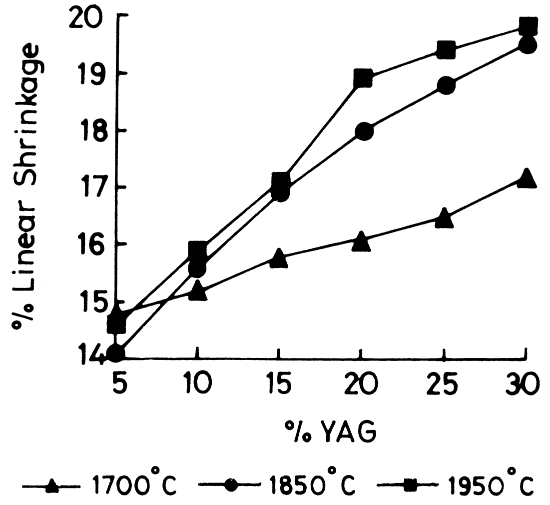

Fig. 14. Linear shrinkage of the specimens in relation to YAG content (set III).

Y-components of the precursors. A large amount of unconverted Al_2O_3 and Y_2O_3 was detected in specimens prepared by mixing oxides of Al and Y and the formation of YAG phase was initiated only around 1400°C, results of which have already been reported [32].

The different phases present in the sintered specimens fired at 1950°C containing 30 wt.% YAG as additive are given in Table 3. In all the systems, SiC and YAG were the major phases present. A moderate amount of $AlYO_3$ was also found in all the samples. Aluminium oxycarbide phases (Al_4O_4C and Al_2OC) in varying amounts were also found with traces of SiO_2. Formation of metasilicate could not be ruled out as SiO_2 was always available as a layer over SiC particles but that could not be detected by XRD analysis due to its amorphous or glassy nature.

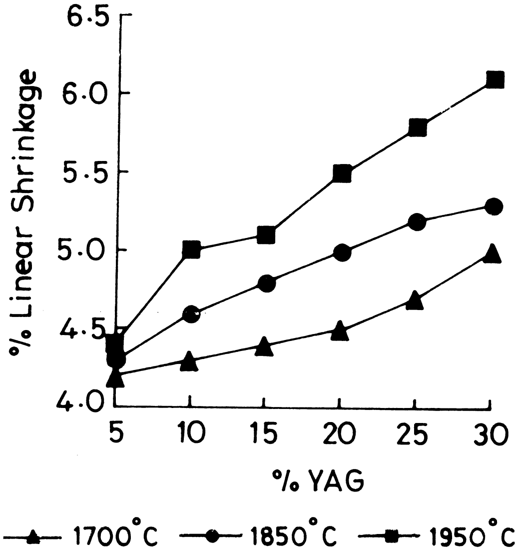

Fig. 15. Linear shrinkage of the specimens in relation to YAG content (set IV).

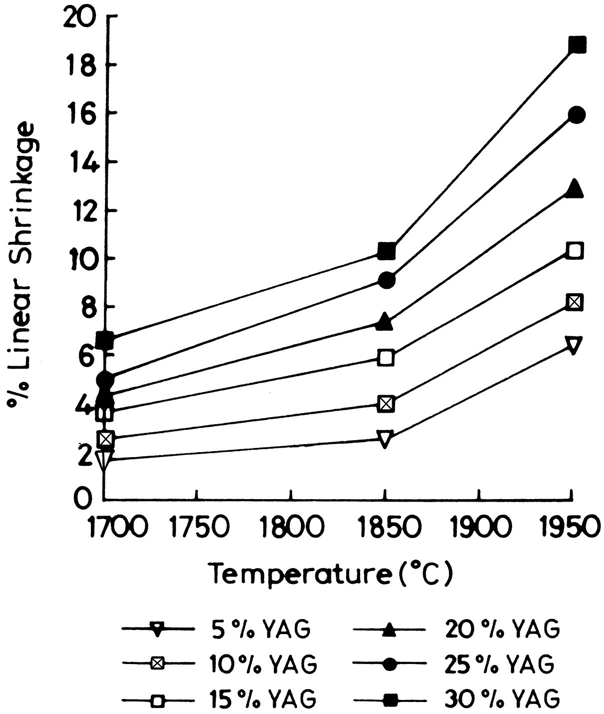

Fig. 16. Linear shrinkage of the specimens with sintering temperature (set I).

4. Discussion

IR studies indicated the presence of –ol and –oxol bonds in the Al–Y–O system even after heat treatment of the hydrogel at 800°C [36] (Fig. 5). This phenomenon increased the reactivity of Al–Y–O system for which YAG was formed at around 800°C as indicated by DTA (Fig. 2) and confirmed by XRD (Fig. 21). Due to the absence of such intermediate bonds (–ol and –oxol),

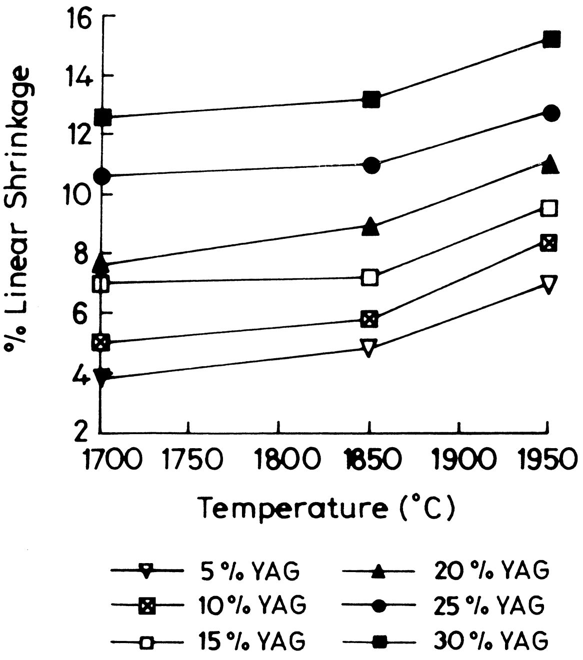

Fig. 17. Linear shrinkage of the specimens with sintering temperature (set II).

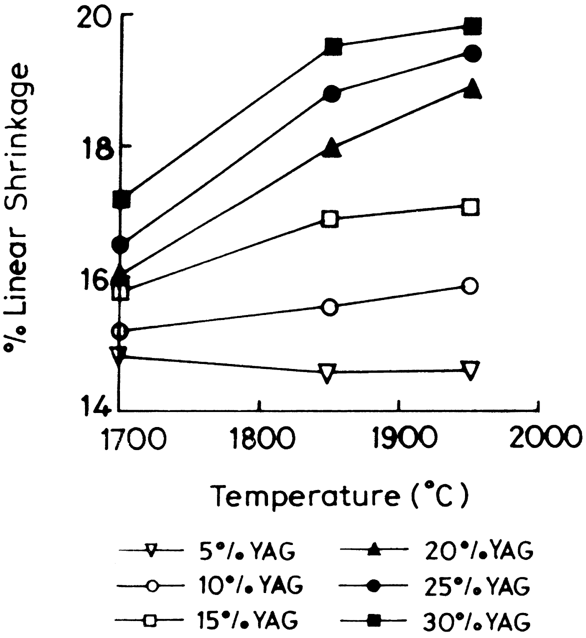

Fig. 18. Linear shrinkage of the specimens with sintering temperature (set III).

reactivity was low when Y_2O_3 and Al_2O_3 were used and where initiation of formation of YAG was at 1400°C[33].

For a general investigation, palletised batches containing SiC with different amount of metal oxides were heat treated in flowing argon atmosphere for 17 h at different temperatures up to 1400°C. The results obtained are shown in Fig. 22. The overall weight loss was small, indicating that the materials were almost completely dehydrated [37,38] during powder preparation stage. Weight loss increased with increasing amount of additive. Small amount of shrinkage of the specimens was followed by comparatively higher shrinkage in the temperature range 1300–1400°C. This was due to the collapse of gel structure.

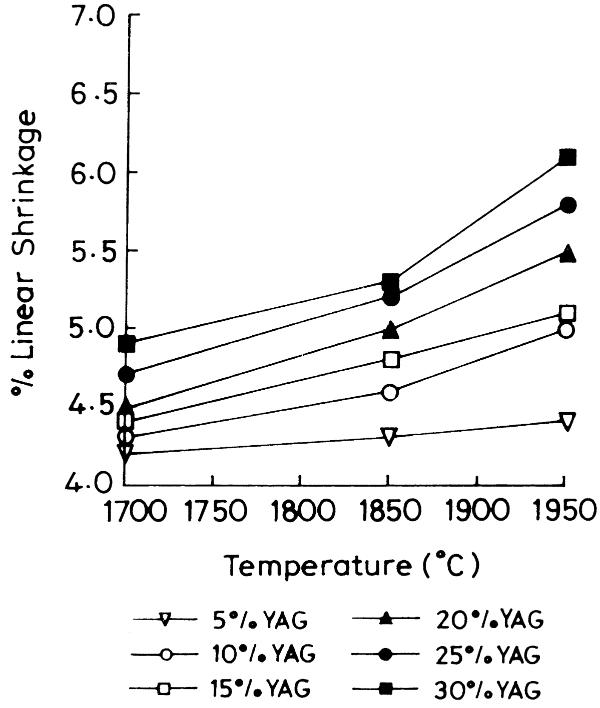

Fig. 19. Linear shrinkage of the specimens with sintering temperature (set IV).

Weight loss in the present system was due to a series of widely reported [39,40] chemical reactions occurring in the range 1400–1950°C as shown below.

- Group 1

$$SiC + 2\,SiO_2 \leftrightarrow 3SiO + CO \tag{1}$$

$$SiC + Al_2O_3 \leftrightarrow Al_2O + SiO + Co \tag{2}$$

$$SiC + Y_2O_3 \leftrightarrow Y_2O + SiO + CO \tag{3}$$

- Group 2

$$SiO + 2C \leftrightarrow SiC + CO \tag{4}$$

$$Al_2O + 3C \leftrightarrow Al_4C_3 + 2CO \tag{5}$$

$$SiC + CO \leftrightarrow 2C + SiO \tag{6}$$

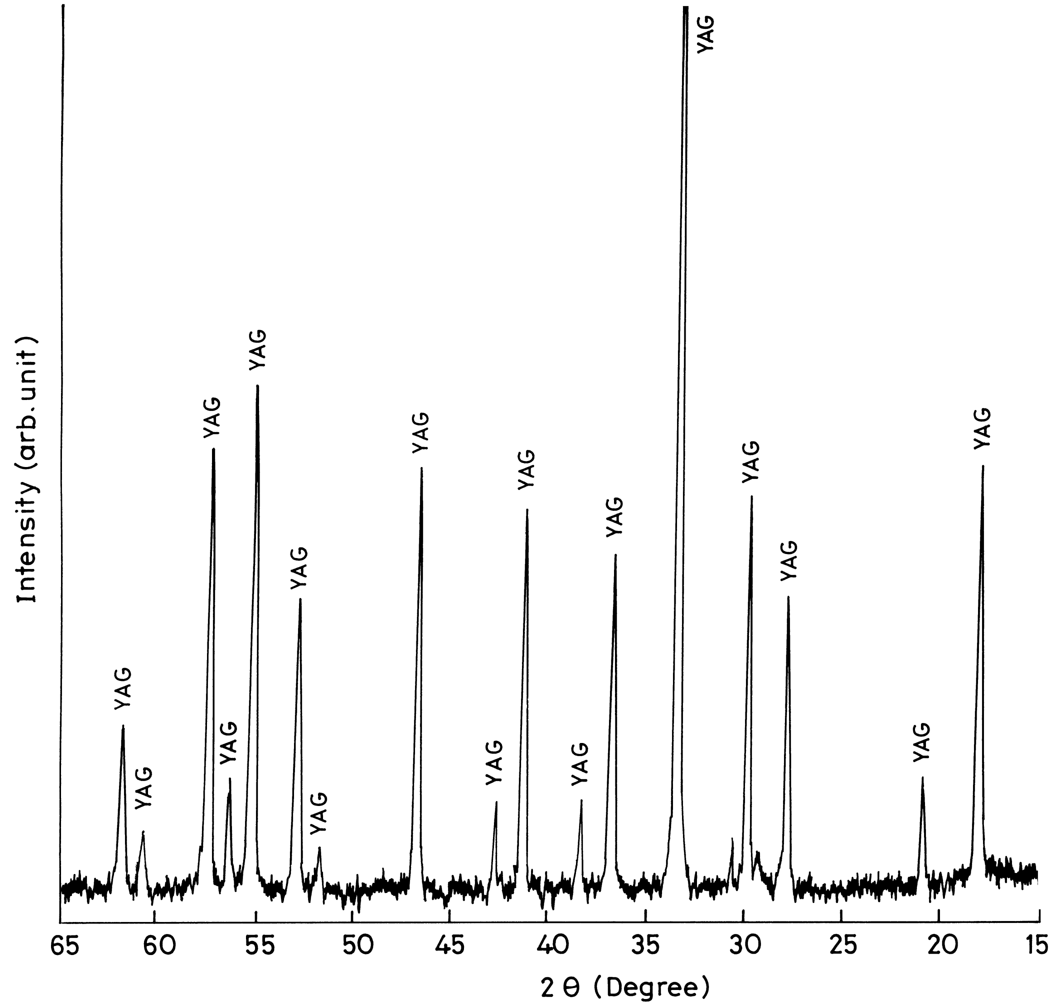

Fig. 20. XRD patterns of Al–Y hydrogel system (Al_2O_3:Y_2O_3 molar ratio 5:3) heat treated at 1400°C for 1 h.

- Group 3

$$Y_3Al_5O_{12} + CO + C \leftrightarrow 3AlYO_3 + 2AlO + 2CO \qquad (7)$$

$$Y_3Al_5O_{12} + 2CO + 2C \leftrightarrow 3AlYO_3 + Al_2O + 4CO \qquad (8)$$

$$Y_3Al_5O_{12} + 4CO + 3C \leftrightarrow 3AlYO_3 + Al_2OC + 6CO \qquad (9)$$

The different volatile species formed by the decomposition reactions were more above 1800°C for set I samples (Fig. 9) and those progressively increased with temperature of heat treatment up to 1950°C. However, when similar specimens had a pre-sintering heat treatment at 1400°C for 2 h, the extent of weight loss was much less. With set II specimens, weight loss was substantially reduced when the sintering temperature was 1950°C (Fig. 10). The highly reactive oxides in set I were responsible for higher decomposition reactions (1)–(6). In general, it was found that weight loss and shrinkage progressed simultaneously by three counteracting mechanisms (a) formation of pores resulting into retarded volume diffusivity, (b) formation of defects [41–43] in the SiC structures with subsequent increase of volume diffusivity and (c) formation of liquid if any. When pre-sintering hold at 1400°C for 2 h was maintained, a lower weight loss was indicative of more stability of the reacting species which was due to the formation of YAG (Fig. 21).

Overall reactivity of SiC towards decomposition reactions with different oxide additives should be more with finer SiC particles. So it may be expected that higher the surface area of SiC the higher would be the dissociation loss, but in the present system weight gain was noted in case of set IV samples in which fine SiC (16 m^2/g) were used. In the set of reactions as shown by Eqs. (1)–(9) weight gain is only possible with reaction (9). Therefore, it may be concluded that increasing fineness of SiC favoured reaction (9). Reactions (7) and (8) contributed towards weight loss of the specimens

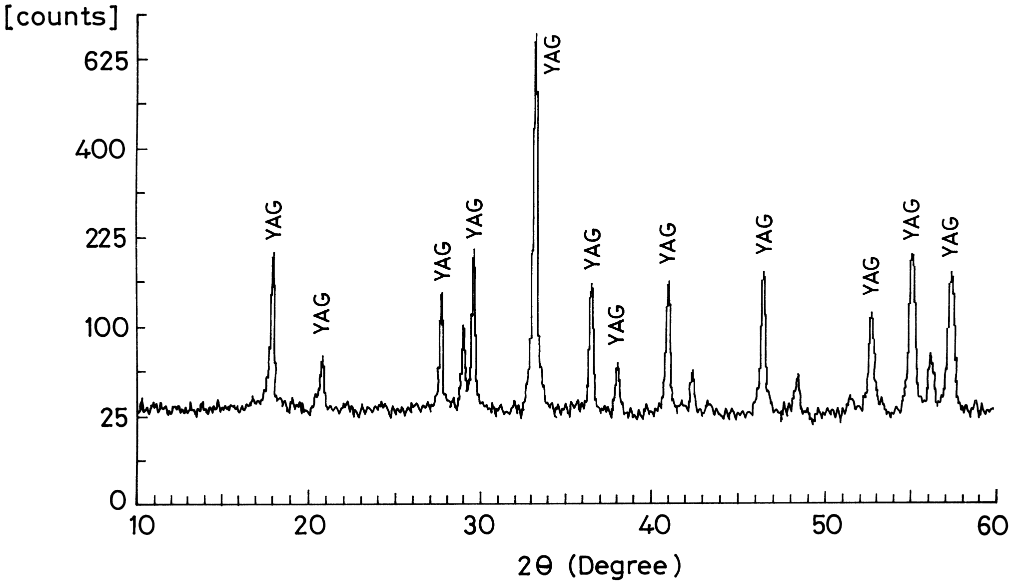

Fig. 21. XRD patterns of Al–Y hydrogel system (Al_2O_3:Y_2O_3 molar ratio 5:3) heat treated at 900°C for 1 h.

which explains the increasing weight loss or in some cases decreasing weight gain of all samples with increasing amount of the additive. Application of pressure (6.33 bar) partially suppressed dissociation reactions responsible for producing volatile reaction products at the sintering temperature [(reactions (1)–(8)].

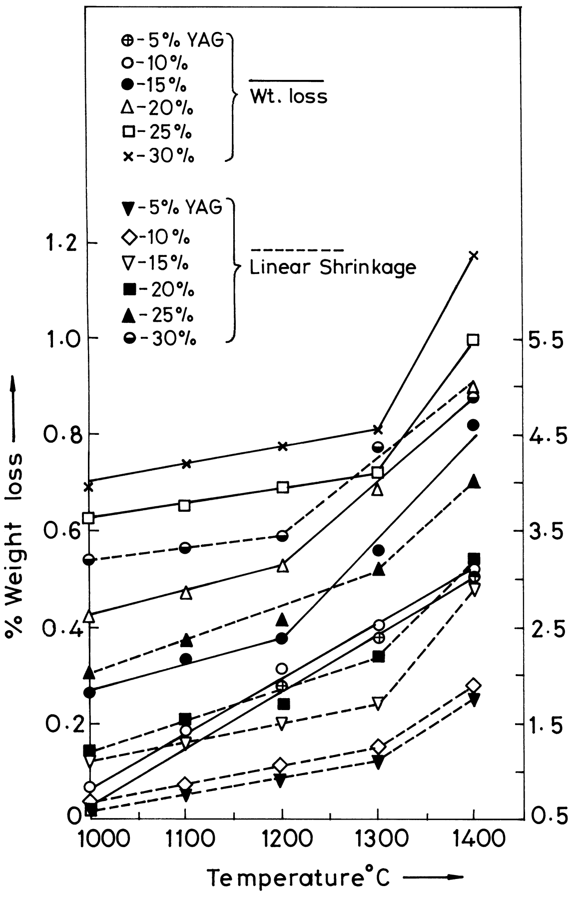

Fig. 22. Weight loss and linear shrinkage of the specimens heat treated in the temperature range 1000–1400°C in argon atmosphere.

Pre-forming of YAG was also found to be responsible for suppression of reactions (1)–(8), as indicated in Figs. 8 and 11. Trace amount of aluminium oxycarbides (Al_4O_4C and Al_2OC) found in sintered samples along with $Y_{4.67}(SiO_4)_3O$ (Table 3) for the samples of sets III and IV (Table 2) supports the above observations.

The overall chemical processes in the present system thus may be summarised as follows: Al_2O_3 and Y_2O_3 in powder bed combined at lower temperature to form YAG and had alike the specimens, sets of similar reactions. SiC and YAG reacted by reactions (7)–(9) in CO atmosphere [44], CO being generated from other reactions. Extent of all those reactions depended on the availability of CO and C, both of which were available in the reaction environment prevailing in the present investigation. The transport of Al_2O_3 from the surroundings (SiC/Al_2O_3/Y_2O_3 in powder bed) into the body during sintering is well established [45]. Transportation of Al_2O_3 from powder bed into specimens through the intermediate formation of Al_2O and AlO was the suggested [46] mechanism which implied a vapour phase transport path [39] between the surroundings and the specimen system leading to weight gain. It should be mentioned here that the above reaction, i.e. transportation of matter from surroundings to the specimen systems which took place throughout the entire process, always led to weight gain. This was counter balanced by the evaporation or dissociation by reactions (1)–(8). The net result was either a loss or a gain of weight of the specimens depending on the

Table 3
Phases present in fired specimens (fired at 1950°C)

Specimen (description)	Sintering conditions	Phases present in fired specimens[a]
Hydrogel derived additive + SiC (coarse)	No hold	SiC (VS),YAG (S), $AlYO_3$ (M), Al_4O_4C (W), Al_2OC (VW), α-Al_2O_3 (W), SiO_2
Hydrogel derived additive + SiC (coarse)	Hold at 1400°C	SiC (VS),YAG (S), Al_4O_4C (M), $AlYO_3$ (M), Al_2OC (W), α-Al_2O_3 (M), Al_4SiC_4 (M), SiO_2
Hydrogel derived additive + SiC (fine)	Hold at 1400°C	SiC (VS), YAG (S), $AlYO_3$ (M), Al_4O_4C (W), α-Al_2O_3 (M), Al_2OC (VW), $Y_{4.67}(SiO_4)_3O$ (W)
YAG + SiC (fine)	No hold	SiC (VS), YAG (S), $AlYO_3$ (M), α-Al_2O_3 (M), Al_4O_4C (W), Al_2OC (VW) $Y_{4.67}(SiO_4)_3O$ (W)

[a] Abbreviations in parentheses indicate the relative peak intensity as: S, strong; VS, very strong; M, medium; W, weak; VW, very weak.

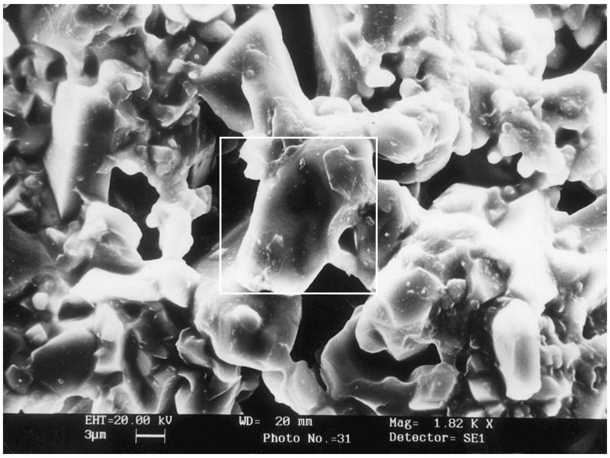

Fig. 23. Scanning electron micrograph of fracture surface (sintered at 1950°C for 0.5 h in Ar, 25 wt.% YAG).

reactions dominating at sintering temperature for the particular system.

SEM analysis of the specimen containing 25 wt.% YAG and sintered at 1950°C for 0.5 h indicated grain growth (Fig. 23). Average equivalent diameter of starting particles increased from 2.89 to $>$ 8 µm. In isolated cases, particles of above 15 µm diameter were also found. About 66% grains were between 6 and 9 µm and the rest were between 10 and 15 µm diameter.

5. Conclusions

1. In-situ formation of YAG by a hold at an intermediate temperature was found to decrease dissociation and enhanced densification and was found to be a better process than addition of preformed YAG or heating the specimens containing yttrium and aluminium oxide as additive without a hold.
2. Dissociation reactions could be suppressed by increasing gas pressure over the reactants and increasing fineness of the SiC used.

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