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High Temperature Phase Equilibrium of SiC-Based Ceramic Systems

Yuhong Chen, Laner Wu ,Wenzhou Sun , Youjun Lu and Zhenkun Huang School of Material Science & Engineering, Beifang University of Nationalities Ningxia, China

1. Introduction

Silicon carbide (SiC) is one of the promising structure materials for mechanical and thermal applications(Nitin P., 1994). Although SiC ceramic has been developed for several decades, it is still important to study in some areas, ally the high temperature phase relations in SiC-based ceramic systems. In addition, the SiC/Si₃N₄ composites are of increasing interest because they should have the complement each other in the mechanical properties.(Kim Y. & Mitomo.M, 2000, Lee Y et.al., 2001) SiC and Si₃N₄ are the strong covalent compounds. The self-diffusion coefficient of Si and C, also Si and N, are too low to get the fully dense ceramics without sintering aids. Rare-earth oxides are often used as liquid phase sintering aids for densification. the behaviours of their high temperature reactions and the derived phase relations are still unknown. Becher (Becher et al, 1996) found that the chemical composition of the grain boundary amorphous phase could significantly influence the interfacial debonding behaviour in silicon nitride. Other study (Keeebe H. et.al., 1996)also showed that the secondary phase chemistry could play a key role in toughening Si₃N₄ ceramic due to its influences on the grain morphology formation, secondary-phase crystallization and residual stress distribution at grain boundaries. For SiC ceramics less of reaction behaviour at high temperature was known due to its sluggish diffusion. About phase relations the Si₃N₄-containing systems have been much published (Anna E. McHale. 1994), but either SiC-based ceramic or SiC/ Si₃N₄ composite systems were rarely done. Even so, the compatibility relations of SiC with neighbour phases should be revealed. Doing so is beneficial to practical use in the manufacture of SiC-based ceramics, as well as SiC/ Si₃N₄ composites.

The present work focused on the determination of the phase relations in the quaternary systems of SiC- Si₃N₄-SiO₂-R₂O₃ (R=La,Gd,Y) at high temperatures. Lanthanum which has lower atomic number in 17 rare earth elements, as a typical light rare-earth oxide, Gd₂O₃ as middle and Y₂O₃ as heavy one with similar property as heavy rare earth oxide were chosen to use in this study. Rare earth oxides used as sintering aids retained in intergranular phases after reaction, which cause strength degradation of the material at high temperature. The investigation of phase relations in this quaternary system will be a summary of work from studies of Si-N-O-R(ANNA E. McHale. (1994)) to Si-C-N-O-R systems. Extensive investigation

for the phase relations and reactives in high temperature is beneficial to practical use in the manufacture of SiC-based ceramics, as well as SiC/ Si_3N_4 composites.

2. Experimental

The starting powders were α -SiC (H.C.Starck), β -Si₃N₄ (H.C.Starck), La₂O₃, Gd₂O₃ and Y₂O₃ (R₂O₃ with 99.9% purity, from Baotou Rare-earth Institute, China). The rare earth oxides were calcined in air at 1200°C for 2h before use. The compositions investigated were restricted to the region bound by the poins SiC, Si₃N₄ and R₂O₃ (R=La,Gd,Y), but SiO₂ came from in situ oxygen impurity on the surface of powders. Selected compositions were made by mixing the required amounts of the starting powders in agate jar mills with absolute alcohol for 2hr. The dried mixtures were hot-pressed in graphite dies 10 mm in diameter lined with BN in a graphite resistance furnace under a pressure of 30MPa at a subsolidus temperature under a mild flow of Ar, as well as N₂ used for comparison. For the systems SiC-R₂O₃, the melting behaviours of SiC and R₂O₃ (1:1 mole ratio) shown in the table 1. In which the subsolidus temperatures were used as the hot-pressing temperatures for some compositions.

R ₂ O ₃ :SiC (1:1)	Temperatures (°C)					
R_2O_3	1600	1700	1750	1800	1850	1900
La_2O_3	not melted	partly melted	melted			
Gd_2O_3		not melted	Little melted	partly melted	melted	
Y ₂ O ₃		not melted	Little melted	Little melted	partly melted	melted

Table 1. Melting behaviors for R_2O_3 : SiC (1:1)

The specimens were hot-pressed for 1 to 2 hr in the high temperature region and then cooled at 200°C/min.. End points of hot-pressing were obtained where no further phase change was observed when specimens were heated for longer times. An automatic recording X-ray diffraction with monochromated CuKa radiation was used to scan the samples at a rate of 2° /min.

3. Phase relation of binary subsystem

3.1 Phase relation of R₂O₃- Si₃N₄ subsystem

Table 2 shows the phase relation for different Si_3N_4 - R_2O_3 binary subsystems in Ar or N_2 atmosphere respectively.

Table 2. phase relation of Si₃N₄-R₂O₃ binary subsystem

In the Y₂O₃- Si₃N₄ subsystem Y₂O₃- Si₃N₄ mililite(M phase) was determined after hotpressing under Ar and N₂ atmosphere. On the M- Y₂O₃ join a richer-oxygen phase, 2 Y₂O₃ Si₂N₂O (J-phase, monocl.) was determined, The binary phase diagram of Y₂O₃- Si₃N₄ under 1MPa N₂ is presented as Fig 1(Huang Z. K. & Tien T. Y.,1996).

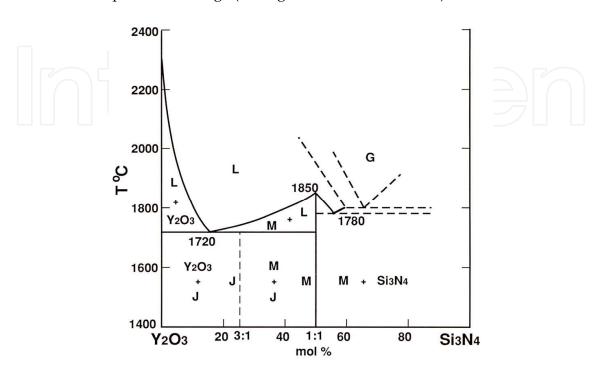
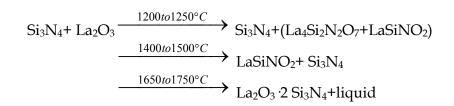


Fig. 1. Phase diagram of Y₂O₃- Si₃N₄ subsystem

The reaction can be written as follows:

 $\begin{array}{l} Si_{3}N_{4}+Y_{2}O_{3} \rightarrow Si_{3}N_{4} \cdot Y_{2}O_{3} \left(Y_{2}Si_{3}N_{4}O_{3}, M\right) \\ Si_{3}N_{4}+SiO_{2}+2 Y_{2}O_{3} \rightarrow 2(2 Y_{2}O_{3} \cdot Si_{2}N_{2}O) \left(Y_{4}Si_{2}N_{2}O_{7}, J\right) \\ The Gd_{2}O_{3}-Si_{3}N_{4} \ subsystem \ has similar \ phase \ relations \ and \ reactions. \\ SiC + Gd_{2}O_{3}+SiO_{2}+3/2N_{2} \rightarrow Gd_{2}O_{3} \cdot Si_{3}N_{4} \ (M \ phase) + CO_{2} \uparrow \\ Si_{3}N_{4}+SiO_{2}+2 \ Gd_{2}O_{3} \rightarrow 2(2 \ Gd_{2}O_{3} \cdot Si_{2}N_{2}O) \ (Gd_{4}Si_{2}N_{2}O_{7}, J) \end{array}$

In the La₂O₃- Si₃N₄ subsystem La₂O₃ ·2 Si₃N₄ (monoclinic 2:1) was determined repeatedly after hot-pressing under either Ar or N₂ atmosphere. A disputed La-melilite (La₂O₃: Si₃N₄) was not found, because of the large radius of La³⁺ ion. It could form only in bigger cell to be La₂O₃. Si₂N₂O. AlN (La₂Si₂ AlO₄N₃, melilite) by Al-N replaced for Si-N(Huang Z. K. & Chen I. W.,1996). LaSiNO₂ (K phase, monoclinic) were determined because of the impurty of powder. On the 2:1- La₂O₃ join a richer-oxygen phase, 2 La₂O₃ Si₂N₂O (J-phase, monocl.) was determined, indicating the presence of excess oxygen from SiO₂ impurity in the powder mixtures. M.Mitomo (Mitomo M.,et.al. 1982)found that an equi-molar mixture of and heated to 1800°C showed that there were three temperature regions in which chemical reaction took place.



3.2 Phase relation of R₂O₃-SiC subsystem

No new phase was detected in SiC- Si₃N₄ and SiC-R₂O₃ (R=La,Gd,Y) systems, it can be due to its very low self-diffusion coefficient of Si and C with very strong covalence of Si-C bond. However , a few of $2R_2O_3$ Si₂N₂O (J phase)was observed in SiC-R₂O₃ system. The oxygen content of SiC powder, existing either as surface SiO₂ or as interstitial oxygen is between 0.8 to 1.1wt%. The reduction of SiC (lower X-ray peak intensity of SiC) indicated that a part of SiC could directly react with R₂O₃ after being oxidized/nitrided under N₂. The reaction can be written as follows:

 $3SiC + 2N_2 \rightarrow Si_3N_4 + 3C$,

 $4R_2O_3 + SiO_2 + Si_3N_4 \rightarrow 2(2R_2O_3. Si_2N_2O)$ (J phase)

It should be noted that only a little amount of oxygen content is enough to form much more rare-earth silicon-oxynitrides as shown below: For the examples of La-siliconoxynitrides, one mole of oxygen can cause formation of 2 moles of J phase (La), $(Si_2N_2O.2La_2O_3)$. It means that 1 wt% O₂ can cause formation of 47.0 wt% J(La) phase.

In fact, it is difficult to make SiC reaction under N_2 , but when rare-earth oxide entered in system, SiC can be reacted even at lower temperature (1550°C for SiC- La₂O₃, 1600°C for SiC-Gd₂O₃ system). The addition of rare-earth oxide benefits the nitride reaction of SiC. Table 3 shows the phase relation in SiC -R₂O₃ binary system in different atmosphere.

ArNo reactionNo reactionNo reaction N_2 J, SiCJ, SiCJ, SiC		SiC- La ₂ O ₃	SiC-Gd ₂ O ₃	SiC-Y ₂ O ₃
N ₂ J, SiC J, SiC J,SiC	Ar	No reaction	No reaction	No reaction
	N_2	J, SiC	J, SiC	J,SiC

Table 3. Formed phase of SiC:R₂O₃=1:1 compositions

4. The phase equilibrium of SiC-Si₃N₄ -R₂O₃

The binary phases of La₂O₃ \cdot 2Si₃N₄ and Si₃N₄.R₂O₃ (M(Gd),M(Y)) coexist with SiC forming a tie-line which separated every ternary system of SiC- Si₃N₄-R₂O₃ (R=La,Gd,Y) into two triangles, respectively. The 2R₂O₃ Si₂N₂O (J phase) also coexist with SiC forming another tie-line in triangle near R₂O₃ side. Based on the experimental results of binary subsystem, the subsolidus phase diagrams of SiC- Si₃N₄-R₂O₃ (R=La,Gd,Y) systems are presented as Fig. 2.Comparing SiC- Si₃N₄-R₂O₃ with AlN- Si₃N₄-R₂O₃ systems (Cao G.Z., et.al,1989) reported by Cao G.Z. et, the similarity is evident except SiC couldn't participate to form α -Sialon because of its tough Si-C bond with bigger bond length 1.89Å.

The XRD pattern of typical sample after hot-pressed of SiC- Si₃N₄ -Y₂O₃ system in N₂ atmosphere is shown in Fig3, phase analysis indicated that M phase (Si₃N₄·Y₂O₃), K phase (Si₂N₂O·Y₂O₃), or J phase (Si₂N₂O·2Y₂O₃) were formed. And in these samples, SiC coexisted with M, K-phase (Fig3-a) , coexisted with Si₃N₄, M-phase(Fig3-b) and with Y₂O₃ ,J phase(Fig3-c). But in sample sintered in Ar atmosphere, K phase had formed instead of J

phase(Fig4). The reason is higher oxygen partial pressure in Ar atmosphere. The introduction of Si₂N₂O transformed the ternary system into the quaternary system. In the system, three compatible tetrahedrons, namely, SiC-M-K-J, SiC-M-J-Y₂O₃, SiC-Si₃N₄-M-K (in N₂) or SiC-Si₃N₄-M-J(in Ar) have been determined. SiC and Si₃N₄ would selectively equilibrate with these three phases in the order of M < K < J < Y₂O₃ with respect to the effects of the oxygen content of SiC and Si₃N₄ powders and the oxygen partial pressure in high temperature. Based on those results, the subsolid phase diagram for the ternary SiC-Si₃N₄-Y₂O₃ system and the quaternary SiC-Si₃N₄-Si₂N₂O-Y₂O₃ system are given in Fig 5.

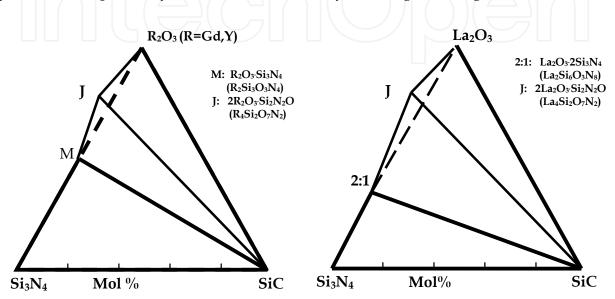


Fig. 2. Subsolidus phase diagram of the system SiC-Si₃N₄-R₂O₃ in Ar or N₂

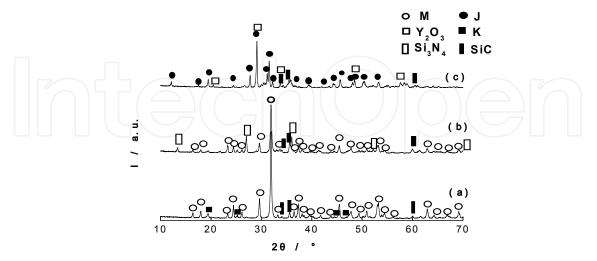


Fig. 3. XRD pattern of SiC-Si₃N₄-Y₂O₃ hot pressed sample in N₂

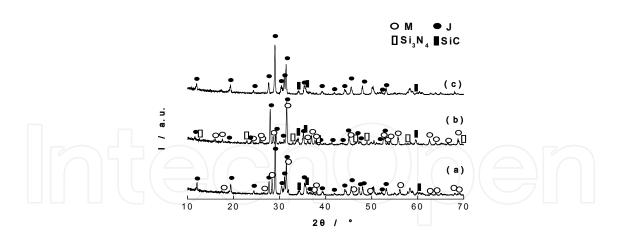


Fig. 4. XRD pattern of SiC-Si $_3N_4$ -Y $_2O_3$ hot pressed sample in Ar

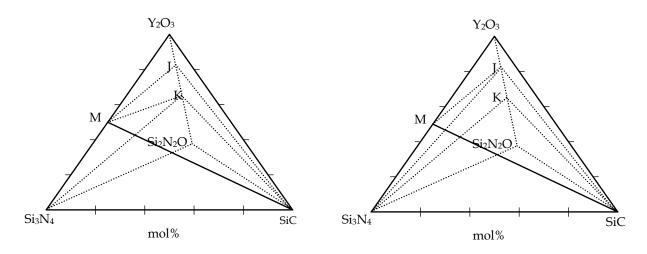


Fig. 5. Subsolidus phase diagram of SiC- Si_3N_4 - Si_2N_2O - Y_2O_3 system(a: in N_2 , b:in Ar

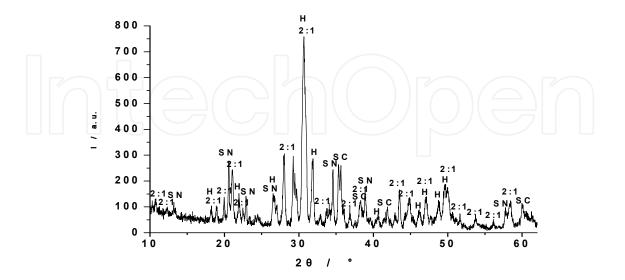


Fig. 6. XRD pattern of SiC-Si $_3N_4$ -2:1-H showing coexistence of four phases in the system SiC-Si $_3N_4$ -La $_2O_3$ -SiO $_2$.

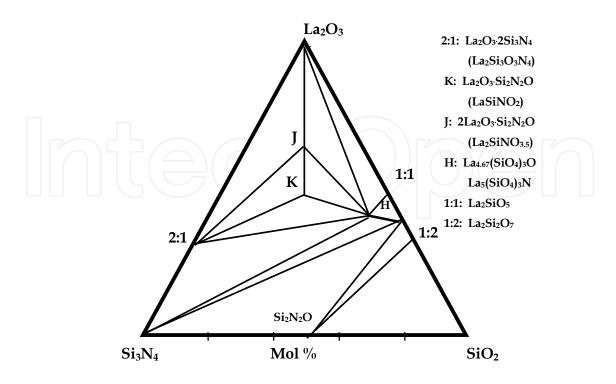


Fig. 7. Subsolidus phase diagram of the system Si₃N₄-SiO₂-La₂O₃ in Ar or N₂^[9,13]

As the typical example, Fig 6 showed XRD patterns of four phase coexistence in two typical tetrahedrons respectively in SiC- Si₃N₄ -La₂O₃ system. The oxygen-richer rare-earth silicon-oxynitrides phase La₅(SiO₄)₃N (H phase) had been indicated in this system. K-phase (Si₂N₂O ·La₂O₃) 2La₂O₃ Si₂N₂O (J-phase) were indicated in this system similar with Si₃N₄ - La₂O₃ system, in which J phase also occurred on the binary composition Si₃N₄:2La₂O₃. It indicates that the formation of above oxynitrides was related to the presence of excess oxygen from SiO₂ impurity in the powder mixtures. It should be noted that these oxygen-richer rare-earth silicon-oxynitrides do not lie on the plane SiC- Si₃N₄-La₂O₃ even so synthesized by these three powders, but lie in the Si₃N₄-SiO₂-La₂O₃ system . The isothermal section at 1700°C of Si₃N₄-SiO₂-La₂O₃ system was reported by M.Mitomo(M.Mitomo,1982). Where he obtained J- and K-phase by crystallization from liquid phase, because they lie by a liquid area. In the present work they were obtained directly by solid-state reaction under hot-pressing at 1550°C and led to construct the subsolidus phase relations of Si₃N₄-SiO₂-

 La_2O_3 system (Fig. 7)(Toropov, et al ,1962, Mitomo, 1982) showing some similarity in both. Above all the oxygen-richer rare-earth silicon-oxynitrides and the three members of ternary systems Si₃N₄-SiO₂-La₂O₃ were compatible with SiC forming ten four-phase compatibility tetrahedrons as follows:

 $\label{eq:sic-si_3N_4-2:1-H, sic-si_3N_4-H-Si_2N_2O, sic-H-Si_2N_2O-1:2, sic-si_2N_2O-1:2-SiO_2, sic-2:1-K-H, sic-2:1-K-J, sic-K-J-H, sic-2:1-J-La_2O_3, sic-J-La_2O_3-H, sic-H-La_2O_3-1:1.}$

The subsolidus phase relationship of this quaternary system with ten four-phase compatibility tetrahedrons is plotted in Fig 8.

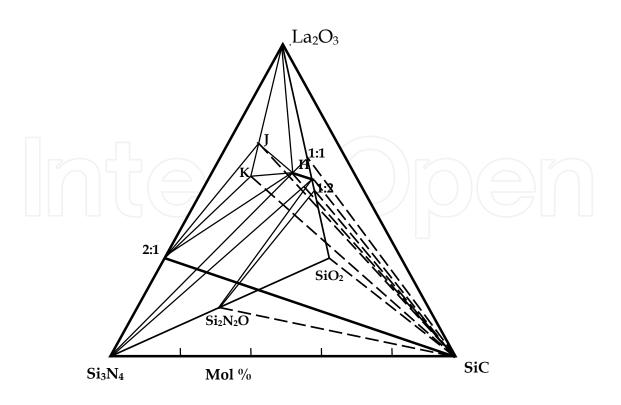


Fig. 8. Subsolidus phase diagram of the system SiC-Si $_3N_4$ -La $_2O_3$ -SiO $_2$ in N $_2$ or Ar

Fig. 8. Subsolidus phase diagram of the system SiC-Si₃N₄-La₂O₃ -SiO₂ in N₂ or Ar In the Si₃N₄-SiC-Gd₂O₃ system, the M-phase(Si₃N₄ Gd₂O₃, J-phase(Si₂N₂O·2Gd₂O₃) and H-phase ($Gd_{10}(SiO_4)_6N_2$) were indicated, a typical XRD pattern of hot-pressure in 1700°C is shown in Fig 9.

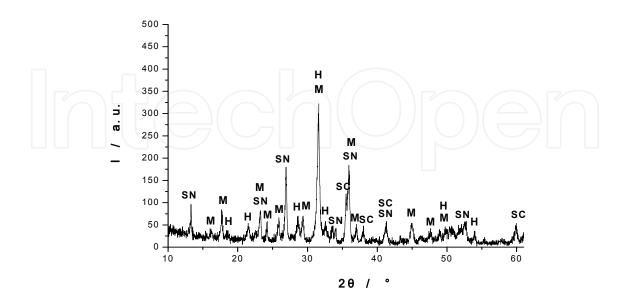


Fig. 9. XRD pattern of SiC-Si $_3N_4$ -M(Gd)-H(Gd) four-phases coexistence in the system SiC-Si $_3N_4$ -Gd $_2O_3$ -SiO $_2$.

Table 4 shows the phase analysis of different compositions in Si_3N_4 -SiC-Gd₂O₃ system. With the increasing of SiC and Si_3N_4 , which means the increasing oxygen content in system, Mphase, J-phase and H-phase would be formed. In the Ar atmosphere, H-phase, which is more oxygen-rich inclined to generation than in N_2 since the higher oxygen particle pressure.

No.	the composition of raw material /mol	Phase composition(in Ar)	Phase composition (in N ₂)
1#	SiC : Si_3N_4 : $Gd_2O_3=4$: 4 : 1	M(vs),Si ₃ N ₄ (s),SiC(m),H(w)	M(vs), Si ₃ N ₄ (s), H(m),SiC(w)
2#	SiC : Si_3N_4 : $Gd_2O_3=1$: 1 : 1	M(vs),J(m),SiC(w)	M(vs),J(m),SiC(w)
3#	SiC : Si_3N_4 : $Gd_2O_3=1$: 1 : 2	J(s),H(m),SiC(w)	J(s),SiC(w)
4#	SiC : Si_3N_4 : $Gd_2O_3=1$: 1 : 4	J(s),Gd ₂ O ₃ (w)	J(vs),SiC(m),Gd ₂ O ₃ (w)

vs: very strong, s: strong, m: middle w: weak

Table 4. The compositions of raw material and phase compositions in ternary systems SiC-Si $_3N_4$ -Gd $_2O_3$ (in Ar or N_2 , 1700°C)

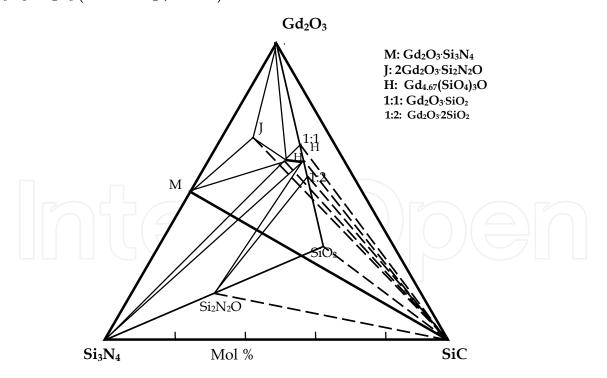


Fig. 10. Subsolidus phase diagram of the system SiC-Si $_3N_4$ -Gd $_2O_3$ -SiO $_2$ in Ar or N $_2$

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The compositions in the triangles bounded by R-SiC tielines and Gd_2O_3 always led to the formation of rare-earth silicon-oxynitrides, indicating the presences of excess oxygen in the powder mixture, that means SiO₂ in powder also participated in the reaction in the system. Presence of SiO₂ leads to the quasiternary system Si₃N₄-SiC-Gd₂O₃ extend into the quaternary system Si₃N₄-SiC-SiO₂-Gd₂O₃. All rare earth silicon-oxinitrides wrer compatible with SiC, forming eight four-phases compatibility terahedrons as follows:

SiC-Si₃N₄-M-H, SiC-Si₃N₄-H-Si₂N₂O, SiC-H-Si₂N₂O-1:2, SiC-Si₂N₂O-1:2-SiO₂, SiC-M-J-H, SiC-M-J-Gd₂O₃, SiC-J-Gd₂O₃-H, SiC-H-Gd₂O₃-1:1,

Hence the subsolidus phase diagram of this quaternary system is plotted in Fig. 10.

5. The high temperature reaction

Generally, the oxygen content of SiC powder, existing either as surface SiO₂ or as interstitial oxygen is between 0.8 to 1.1wt%. More than 1.5% of oxygen content exists in Si₃N₄ powder. The in-situ SiO₂ coexisting with powder mixture leads to the quasiternary systems SiC-Si₃N₄-R₂O₃ extend into the quaternary systems SiC-Si₃N₄-SiO₂-R₂O₃ (R=La,Gd,Y).Just as discussed, only a little amount of oxygen content is enough to form much more rare-earth siliconoxynitrides. That is the reason for easier and much more formation of oxygen-richer rare-earth siliconoxynitrides in the present systems. Their formations are essentially based on the reactions of SiO₂ and Si₃N₄ with R₂O₃, but without Si₂N₂O presence as following:

The formation of oxygen-richer rare-earth siliconoxynitrides are often accompanied with not only consuming Si_3N_4 but also reducing SiC (much lower X-ray peak intensity of SiC) specific when hot-pressing under N₂ atmosphere. This implies that a part of SiC could also directly react with R₂O₃ after being oxidised/nitrided. A few of ₂R₂O₃ Si₂N₂O were observed from SiC-R₂O₃ binary system when firing in N₂ atmosphere. In this case the reactions of SiC and R₂O₃ can be written as follows:

SiC + $O_2 \rightarrow SiO_2 + C$, $4SiO_2 + 2N_2 \rightarrow 2Si_2N_2O + 3O_2$; $4SiC + O_2 + 2N_2 \rightarrow 2Si_2N_2O + 4C$; $3SiC + 2N_2 \rightarrow Si_3N_4 + 3C$, then $2SiC + 2R_2O_3 + N_2 + 1.5O_2 \rightarrow 2R_2O_3 Si_2N_2O$ (J phase) + 2CO. $2SiC + R_2O_3 + N_2 + 1.5O_2 \rightarrow R_2O_3 Si_2N_2O$ (K phase) + 2CO. $6SiC + 5R_2O_3 + N_2 + 7.5O_2 \rightarrow 2(R_5(SiO_4)_3N)$ (H phase) + 6CO. $3SiC + R_2O_3 + 2N_2 \rightarrow R_2O_3$. Si₃N₄ (M phase) + 3C.

Table 5 summarizes the formation of rare-earth silicon-oxynitrides in the present systems, indicating the trend of formation lessens with decreasing bond ionicity from SiO₂ to SiC.

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	Ionicity	La_2O_3	Gd_2O_3	Y_2O_3
SiO ₂	5	2:1,H*,1:1	2:1,H*,1:1	2:1,H*,1:1
Si_2N_2O	4#	J(1:2),K(1:1),H**	J(1:2),H**	J(1:2) ,K(1:1),H**
Si_3N_4	3	2:1	M(1:1)	M(1:1)
SiC (in Ar)	2	No	No	No
SiC (in N ₂)##	2	J	J	J
*H : R _{4.67} (SiO ₄) ₃ O. **H: R ₅ (SiO ₄) ₃ N or		Si ₂ N ₂ O.		

Ionicity of Si_2N_2O : 5 for Si-O bond, 3 for Si-N bond.

##A few of J phase formed.

Table 5. Formation of some rare-earth siliconoxynitrides (mole ratio)

6. Conclusion

Subsolidus phase diagrams of the ternary systems SiC- Si₃N₄-R₂O₃ (R=La,Gd,Y) were determined. The in-situ SiO₂ impurity in the powder mixtures leads to form some oxygen-richer rare-earth siliconoxynitrides and extend the quasiternary systems into quaternary system of SiC-Si₃N₄-SiO₂-R₂O₃. The phase relations of these quaternary systems were established with several SiC-containing four-phase compatibility tetrahedrons. The formation of oxygen-richer rare-earth siliconoxynitrides was discussed. When firing under nitrogen atmosphere a part of SiC could also directly tend to react with R₂O₃ after being oxidised/nitrided forming some rare-earth siliconoxynitrides. They all contributed to construct the phase diagrams of quaternary systems SiC-Si₃N₄-SiO₂-R₂O₃.

Acknowledgements

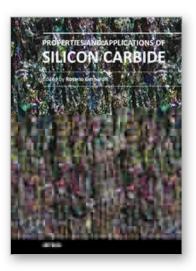
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In this book, we explore an eclectic mix of articles that highlight some new potential applications of SiC and different ways to achieve specific properties. Some articles describe well-established processing methods, while others highlight phase equilibria or machining methods. A resurgence of interest in the structural arena is evident, while new ways to utilize the interesting electromagnetic properties of SiC continue to increase.

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