

High temperature sintering of SiC with oxide additives: IV. Powder beds and the influence of vaporization on the behaviour of SiC compacts

S. Baud^{a,*}, F. Thévenot^a, C. Chatillon^b

^a*Ecole Nationale Supérieure des Mines de Saint-Etienne, 158 cours Fauriel, 42023 Saint-Etienne Cedex 2, France*

^b*Laboratoire de Thermodynamique et Physico-Chimie Métallurgiques (INPG/UJF/CNRS UMR 5614),
ENSEEG, BP 75, 38402 St Martin d'Hères, France*

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Abstract

The liquid phase sintering of SiC with Al₂O₃–Y₂O₃ additives is performed at high temperature and some loss of alumina is mainly observed. Powder beds made of SiC + Al₂O₃ coarse grains are used to counteract these losses. The present mass spectrometric study is aimed at the transfer of matter by the gas phase between the powder bed and the previously sintered pellet. Partial vapor pressures of Al(g), Al₂O(g), SiO(g) and CO(g) are compared over the pellet alone, the beds alone and for the (pellet + bed) mixtures. Those of Al(g) and Al₂O(g) are only slightly modified when the SiC powder is from different origins, meanwhile the CO(g)/SiO(g) ratio is very sensitive to the nature of the SiC powder. Thus, the main effect of the powder bed is not only to prevent or to limit the alumina losses, but also to impose carbon and oxygen activities for the sintering process. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Mass spectrometry; Powder beds; SiC (Al₂O₃–Y₂O₃); Sintering; Vaporisation

1. Introduction

SiC sintering is usually performed at very high temperature ~2200 °C with some B₄C–C additives. In order to decrease this sintering temperature, a liquid phase has been tentatively used, the main components of this liquid being Al₂O₃–Y₂O₃ mixtures, the composition of which is close to the eutectic in the YAG–Al₂O₃ range. In this case, temperature could be decreased to the 1800–2000 °C range, but simultaneously the ceramists observed large weight losses of the SiC pellets and/or additive compounds.^{1–6} In order to decrease these weight losses, a buffer bed of SiC large grains + Al₂O₃ powders is used, either disposed as alternate layers or as mixed powders, that surround the green compacts of SiC + additives to be sintered.^{1,2,4–6} Weight losses or gains may be observed^{4,5} for the compacts. Moreover, the whole system is usually placed in a graphite vessel, closed by a cover in which small holes are drilled for initial pumping before filling with Argon.

Owing to the high temperature working of this sintering process, the weight losses are attributed to vaporization of the mixtures SiC + Al₂O₃ used as buffer beds that are accompanied by matter exchanges between the bed and the compacts. Rather simple explanations of these losses have tentatively been made based on a priori chosen elementary chemical reactions,^{1,7–9} but their conclusions are largely inaccurate because they do not take into account the numerous actual reactions that occur in the quaternary—Al–C–O–Si—system.¹⁰ For these reasons we first have performed a thermochemical analysis of the behavior of the SiC–Al₂O₃ pseudobinary in the whole Al–C–O–Si quaternary system, taking into account the impact of matter losses by the gas phase.¹¹

As shown in our thermodynamic analysis,¹¹ the vaporization of the beds should be pseudo-congruent, i.e. the gas flow composition is included in the SiC–Al₂O₃ pseudobinary section.

Consequently, any matter loss by the gas phase—like effusion under vacuum or diffusion or convective flow in argon through any orifice—will not draw the solid composition out of the pseudo-binary section SiC–Al₂O₃. For starting compositions rich in SiC, the compositional

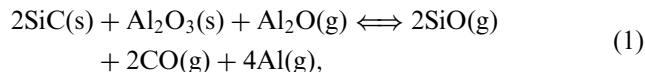
* Corresponding author.

E-mail address: chatillo@ltpcm.inpg.fr (S. Baud).

evolution of the beds is towards richer SiC ones. We observed also that at higher temperatures than 2100 K, liquid silicon or Si–Al alloys can be produced as a consequence of vaporization losses because the SiC–Al₂O₃ mixture is no longer pseudo-congruent.

When sintering SiC–Al₂O₃–Y₂O₃ compacts, in an Al₂O₃–SiC bed, the thermodynamic activity of Al₂O₃ in the compact should be lower than in the SiC–Al₂O₃ bed since the liquid Al₂O₃–Y₂O₃ phase composition close to the eutectic YAG–Al₂O₃ composition is easily formed^{1,12} at the beginning of the heating process, and the alumina activity should decrease. Consequently some Al₂O₃ transfer should occur from the bed to the sample. This transfer competes with the whole vaporization process of the compacts and their bed, and the enrichment with alumina is not always observed, especially for long sintering times.

A first qualitative analysis by quadrupole mass spectrometry of the gas emitted under vacuum from a tungsten substrate on which the two powders (SiC and alumina—YAG) were loaded without any contact,¹³ revealed that molecules at masses 43 and 44 were present. These masses were attributed to AlO(g) and SiO(g) and/or CO₂(g) gases. In the course of our study, the main vaporization reaction as predicted by thermodynamics¹¹ has been confirmed by mass spectrometry using a Knudsen cell¹⁴ to be actually



meanwhile a complete analysis of the ionization patterns proved that AlO(g) and CO₂(g) could not be present in the gas phase at an appreciable level ($<10^{-4}$). The relative proportions of the observed molecules Al₂O(g), SiO(g), CO(g) and Al(g) were close to the one in reaction (1). A quantitative analysis of the partial vapour pressures showed that reaction (1) does not occur at equilibrium because the vaporization is kinetically hindered.¹⁵ The vaporization kinetics occurring in our Knudsen-cells has been characterized by the determination of the evaporation and condensation coefficients of each gaseous species at the surface of the bed. The main effect of these coefficients is to shift the steady-state partial pressures in the quite closed vessel—i.e. the Knudsen cell—from those calculated at equilibrium. Depending on the relative values of the evaporation (α) or condensation (β) coefficients, the steady-state partial pressure of a gaseous species may be higher ($\beta < \alpha$) or lower ($\beta > \alpha$) than the equilibrium one.¹⁵

As these coefficients are specific to the gaseous species and the encountered solid surfaces, their value may be different for the powder beds or for the compacts. Consequently, the influence of kinetically hindered vaporization of the beds and of the compacts may be to completely disturb the matter exchange between the

beds and the compacts as expected from thermodynamics. In order to check this matter exchange, we use a multiple Knudsen-cell mass spectrometric device (Fig. 1) as in preceding papers^{14,15} to intercompare directly the partial pressures for the vaporization of the compacts, the powder beds, and the compacts with their powder beds. The powder beds were from two different origins—i.e. SiC carborundum and SiC norton (see Ref. 14)—for which different sintering characteristics have been observed.^{4,5}

2. Experimental

If the sintering conditions are quite similar to the Knudsen-cell geometry and materials—i.e. quite closed vessel, machined in dense graphite (ET 10, Albertoenis, density 1.75, grains 10 μm , porosity 8%, 0.1% ash residue), covered with a compact SiC layer after one or two experiments¹⁴—the temperature range for mass spectrometric analysis is lower, i.e. 1300–1700 K, because the molecular flow conditions at the effusion orifice requires a total pressure lower than 10^{-4} atm. Consequently, the high temperature stage of the sintering

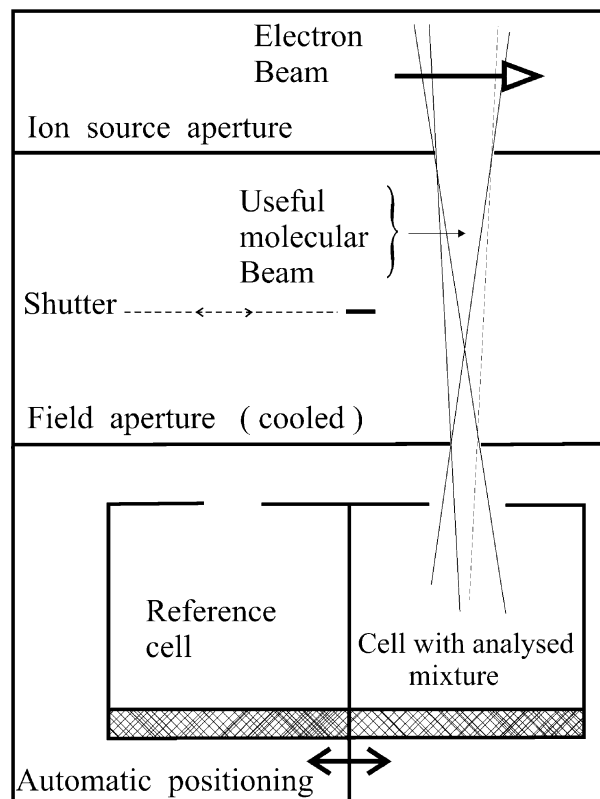


Fig. 1. Principle of the multiple Knudsen cell measurements: four cells (in our case) are disposed in an isothermal envelope, heated in a furnace. The different molecular beams, issued from the four effusion orifices, are successively turned in front of the ion source of a mass spectrometer, and the ionic intensities, proportional to partial pressures are measured and directly compared.

process cannot be analyzed directly in one run, and the sintered compacts must be prepared separately before loading them in the Knudsen cells, with or without their powder beds.

Dense pellets of SiC (83 weight%) and Al₂O₃–YAG eutectic composition (17 weight%) sintered at 1950 °C under Argon (1 bar) during 3 h and a half were used (resulting density ~0.97–0.98 theoretical one, with no open porosity). The upper surface of these pellets is polished and placed in the Knudsen cell with the polished surface facing the effusion orifice. Some of these pellets were covered with the powder bed, SiC + 15 wt.% Al₂O₃. As already explained in the preceding papers,^{14,15} two different SiC powders—carborundum and norton—are used (carborundum SiC with 0.12%O, norton SiC with 0.24%O). The small oxygen content is quickly vaporized (and lost at the beginning of the experiments) as shown in our first mass spectrometric work when dealing with impurities.¹⁴ In order to intercompare directly the vaporization behavior of different samples, we use the multiple cell method¹⁴—i.e. four cells are disposed together in an envelope and loaded in the furnace assembly (Fig. 1). The cells characteristics are presented in Table 1. The main feature of mass spectrometry is then to compare the different ionic intensities of the same species as measured for the different samples, these intensities being directly proportional to their partial pressures in the cells. The intercomparison in the same run does not need any calibration procedure for the mass spectrometer and the absolute values of the partial pressures are not needed, conversely to what was done in a preceding study of the vaporization enthalpy of reaction (1).¹⁵

3. Mass spectrometric results

3.1. The SiC carborundum bed–pellet interaction

The matter exchange between the bed and the pellet is analysed when comparing the partial pressure ratios p_i

(Carb. bed)/ p_i (pellets) and p_i (Carb.bed)/ p_i (pellets + Carb. bed), that is the p_1/p_2 and p_1/p_3 according to Table 1, and this is for the four gaseous species Al, Al₂O, CO and SiO since the pressures of the species Y and YO are too small to be observed (see Ref. 11).

The first ratio p_1/p_2 intercompares the vaporization of the lone bed and the lone pellet. As shown in Fig. 2 for SiO(g) and Al(g), [the Al₂O(g) and CO(g) behavior are similar to SiO(g)], we observe a time interval to reach a steady-state (1 h for SiO, 3 h for Al), and some evolution after 5 h of effusion for SiO(g). The same features were observed in preceding studies^{14,15} due to (i) impurity losses, mainly excess SiO₂ through SiO(g), and (ii) changes in the contact numbers between grains in the powder bed due to preferential vaporization of the smaller grains. Mean values of these ratios are reported in Table 2 (first line), discarding initial or extreme values. The same observations for the (pellet + Carb.

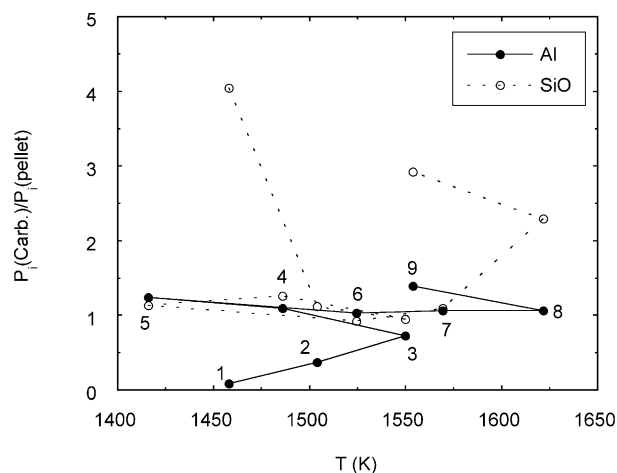


Fig. 2. Evolution with temperature of the partial pressure ratios of Al(g) and SiO(g) between two cells (see Table 1) loaded with a carborundum powder bed and with the lone pellet (already sintered). The quoted numbers refer to the successive temperature plateaus as done during the multiple cell experiment (although the experiment duration is several hours, it is not a time scale).

Table 1
Multiple cell experiment characteristics; materials, samples, crucibles and lids as used

Cell	Sample	Cell material	Effusion orifice ^a (mm)	Partial pressures nomenclature
1	SiC (carborundum) + 15% (weight) Al ₂ O ₃ powder bed	SiC coated Graphite	$h=2$ $d=2$	p_i (Car)
2	Sintered pellet	SiC coated Graphite	$h=2$ $d=2$	p_i (pellet)
3	SiC (carborundum) + 15% (weight) Al ₂ O ₃ powder bed + sintered pellet	SiC coated Graphite	$h=2$ $d=2$	p_i (Car + pellet)
4	SiC (norton) + 15% (weight) Al ₂ O ₃ powderbed + sintered pellet	SiC coated Graphite	$h=2$ $d=2$	p_i (Nor + pellet)

^a h is for cylindrical orifice length, d for diameter.

The original graphite cells were soon covered with a SiC dense layer after the first run of the series (not quoted here).

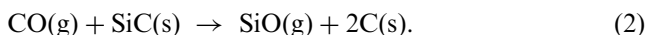
Table 2

Mean values and standard deviations in the 1400–1600 K range of partial pressures ratios between cells loaded with different samples (some values are discarded: see text)

Gaseous species	Al	Al ₂ O	CO	SiO
$\frac{p_i(\text{carb. bed})}{p_i(\text{pellet})}$	1.14 ± 0.13	1.6 ± 0.7	4.21 ± 0.58	1.08 ± 0.11
$\frac{p_i(\text{carb. bed})}{p_i(\text{carb. bed} + \text{pellet})}$	1 ± 0.04	1.02 ± 0.12	1.31 ± 0.35	0.78 ± 0.15
$\frac{p_i(\text{pellet})}{p_i(\text{carb. bed} + \text{pellet})}$	0.88 ± 0.11	0.64 ± 0.29	0.31 ± 0.1	0.72 ± 0.16

bed) compared with the bed behavior are shown in Fig. 3 and summarized in Table 2 (second line). In this case, the time to reach the steady-state is 3 h for Al and Al₂O, meanwhile CO and SiO seem quite stable. We report in Table 2 also the ratio between the lone pellet and the (pellet + Carb. bed). The relative position of pressure values, in arbitrary units, between the lone bed, the lone pellet and the mixture are presented in Fig. 4 for each species.

For the dense pellet and the SiC Carborundum bed, the vaporization kinetics are the same for SiO(g), and quite similar for Al(g) and Al₂O(g), meanwhile the vaporization kinetics of CO(g) is clearly smaller for the pellet. In Fig. 4, we observe that the partial pressures of the species Al, Al₂O and SiO over the (bed + pellet) are higher than for the lone bed or the lone pellet, that means there exists a production of this species in the bed + pellet assembly. Conversely, if the partial pressure over the bed + pellet and/or the lone pellet, is lower than over the lone bed the species is consumed, which is the case for CO(g). Thus, we can conclude that the pellet consumes the CO(g) issued from the bed, meanwhile SiO(g) is produced, which corresponds to the reaction,



X-ray diffraction measurements of the bed and the pellet after experiment showed that C is effectively created in the bed. We observed the same phenomenon¹⁵ for the lone bed, but for longer effusion time i.e. higher mass loss: these observations show that the pellet accelerates the C production in the Carborundum bed by consumption of CO(g).

3.2. The SiC norton bed–pellet interaction

In principle, the same experiment can be performed with the SiC norton–alumina powder bed, but we can use secondary compositions or samples to intercompare the behaviors of the Norton beds during our experiments.^{14,15} Thus the $p_i(\text{Nor.})/p_i(\text{Nor.} + \text{pellet}) = p_i(\text{Nor.})/p_i(\text{Carb.}) \times p_i(\text{Carb.})/p_i(\text{Nor.} + \text{pellet})$, with the assumption that the pressure variations (which are not very important in the Knudsen cells, i.e. some%) of any bed are almost the same when comparing beds with 15 and

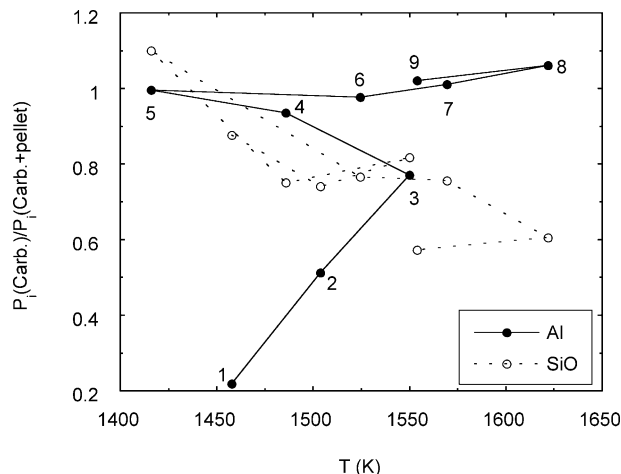


Fig. 3. Evolution with temperature of the partial pressure ratio of Al(g) and SiO(g) between two cells (see Table 1) loaded with the carborundum powder bed and with the (pellet + carborundum bed). The quoted numbers refer to the successive temperature plateaus as done during the multiple cell experiment (it is not a time scale).

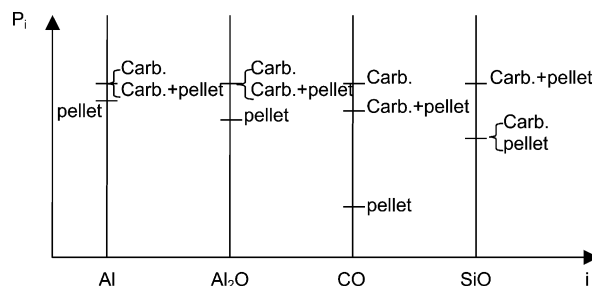
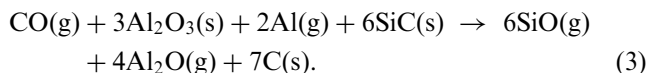


Fig. 4. Qualitative representation from Table 2 of the relative positions of partial pressures of the four main gaseous species according to the different samples loaded in the same cells. Samples refer to the lone carborundum bed (Carb.), the lone pellet or their combination (pellet + bed).

71.8% (weight) of alumina, are presented in Table 3. The relative pressure values between the different cells, loaded with the lone pellet, the lone Norton bed and the (pellet + norton bed) are presented in Fig. 5. The vaporization kinetics of Al(g), Al₂O(g) and SiO(g) are more important for the pellet than for the lone bed, and conversely for CO(g). According to the same reasoning as in the preceding part, CO(g) produced by the Norton powder bed is consumed by the pellet, meanwhile Al(g), Al₂O(g) and SiO(g) are produced by the pellet, especially SiO(g). The reaction occurring may be,



As we did not observe by X-ray diffraction any trace of C(s) either in the pellet or in the powder bed, we cannot confirm reaction (3). This feature conversely may prove that the vaporization reaction remains pseudo congruent or pseudo azeotropic according to the

Table 3

Mean values and standard deviations in the 1400–1600 K range of partial pressures ratios between cells loaded with different samples: carborundum bed, norton bed, pellet or beds + pellet

Gaseous species	Al	Al ₂ O	CO	SiO
$\frac{p_i(\text{carb bed})}{p_i(\text{nor bed+pellet})}$	1.05±0.24	1.07±0.38	9.6±2.7	0.11±0.02
$\frac{p_i(\text{nor bed})}{p_i(\text{carb bed})}$	0.55±0.05	0.46±0.06	0.51±0.09	0.69±0.11
$\frac{p_i(\text{nor bed})}{p_i(\text{nor bed+pellet})}$	0.58±0.14	0.49±0.19	4.9±1.63	0.08±0.02
$\frac{p_i(\text{nor bed})}{p_i(\text{pellet})}$	0.63±0.09	0.74±0.34	2.15±0.48	0.75±0.14
$\frac{p_i(\text{pellet})}{p_i(\text{nor bed+pellet})}$	0.92±0.24	0.67±0.38	2.28±0.71	0.10±0.02

main reaction (1), the partial pressure being modified by the evaporation and condensation coefficients in such a manner that the congruent composition of the gas flow remains in the pseudobinary section SiC–Al₂O₃, as already analysed.¹¹

3.3. Comparison of the two powder beds behavior

As shown in the quantitative vaporization study,¹⁵ and in the preceding parts, the two beds—norton and carborundum—produce different partial pressures either alone or in the presence of the pellets. The relative behavior of the two beds is summarized in Fig. 6 and we observe that for Al(g) and Al₂O(g) their behaviors are similar and close to the lone pellet. Contrarily, for CO(g), and SiO(g) there exists opposite and large evolutions. The largely different values observed for the

ratio $p_{\text{SiO}}/p_{\text{CO}}$, added to similar $p_{\text{Al}}/p_{\text{Al}_2\text{O}}$, show that at least one of the mixtures (bed + pellet) cannot vaporize congruently. The Carborundum bed effectively produced some C(A), but nothing was observed for the Norton one.

In order to understand the exchange of matter between the powder bed and the pellet, we analyzed also the pellets before and after the experiments by X-ray diffraction (Fig. 7). The sintered pellet before vaporization in the effusion cell shows some peaks of a secondary phase, not clearly identified, and these peaks disappeared after effusion with a norton bed, confirming the evolution towards a congruent behavior. This is not the case for the pellet in the carborundum bed.

In addition, the weight losses of the pellet and (pellets + beds) (Table 4) during the effusion experiments confirm the weight losses observed when sintering: the smaller weight loss obtained with the Carborundum bed as compared with the lone pellet weight loss proves that the Carborundum bed transfers alumina to the pellet.

From Tables 2 and 3 we can deduce the relative variation of the CO(g)/SiO(g) pressure ratio, referred to the lone pellet, consequent on the use of the different powder beds by calculating the following ratios,

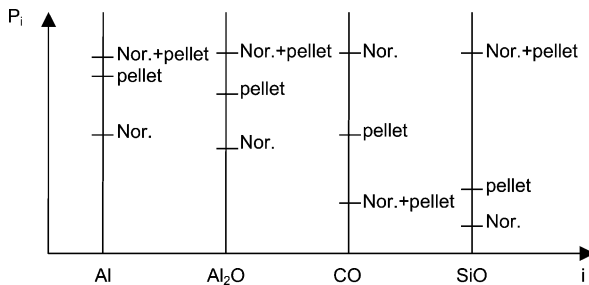


Fig. 5. Qualitative representation from Table 3 of the relative positions of partial pressures of the four main gaseous species according to the different samples loaded in the same cells. Samples refer to the lone Norton bed, the lone pellet or their combination (pellet + bed).

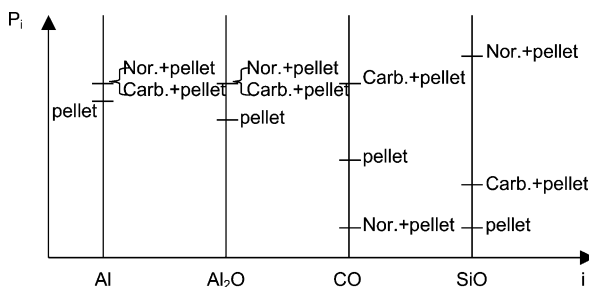


Fig. 6. Qualitative representation of the relative positions of partial pressures of the four main gaseous species as deduced from experiments when using the two different SiC beds: carborundum or norton.

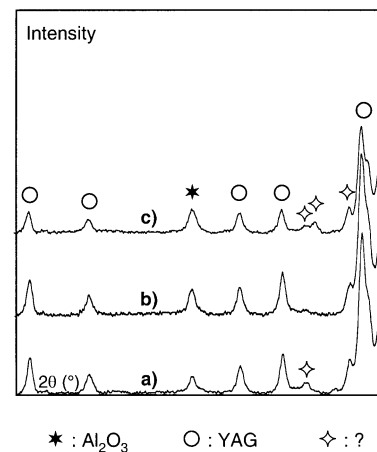


Fig. 7. X-ray diffraction patterns of dense sintered pellets: (a) before vaporization in an effusion cell, (b) after vaporization in a norton + alumina powder bed, (c) after vaporization in a carborundum + alumina powder bed.

Table 4
Mass loss of the different pellet samples during a multiple cell effusion mass spectrometric experiment

	Cell. 2	Cell. 3	Cell. 4
SiAlY-01M experiment	Dense pellet	SiC (carborundum) + 15%(weight) Al ₂ O ₃ powder bed + dense pellet	SiC (norton) + 15%(weight) Al ₂ O ₃ powder bed + dense pellet
Δm Pellet mass loss (%)	−0.3	−0.03	−13.1

$$\frac{p(\text{Carb} + \text{pellet})/p(\text{pellet})}{\left(\frac{p(\text{Nor} + \text{pellet})}{p(\text{pellet})}\right)_{\text{CO or SiO}}}$$

as presented in Table 5. We observe that the CO(g)/SiO(g) pressure ratio over the Carborundum bed with the pellet is about 50 times than over the Norton bed with the pellet.

3.4. Micrographic observation of pellets

The three pellets were observed after effusion by scanning electron microscopy (Fig. 8). The edge of SiC pellet grains evaporated with the norton bed are smooth

Table 5
Experimental CO/SiO pressure ratios as observed for pellets in (carborundum or norton/SiC + alumina) beds

Pressure ratios	Gaseous species		
	CO	SiO	CO/SiO
$\frac{p(\text{carb} + \text{pellet})}{p(\text{pellet})}$	3.22	1.39	2.32
$\frac{p(\text{nor} + \text{pellet})}{p(\text{pellet})}$	0.44	10	0.045

and well defined by a clear contrast, whereas the intergranular phase Al₂O₃–Y₂O₃ appears partly evaporated. The micrographs show that the evaporation of the intergranular phase is non regular and we believe this is depending on the relative concentration of Al₂O₃ and

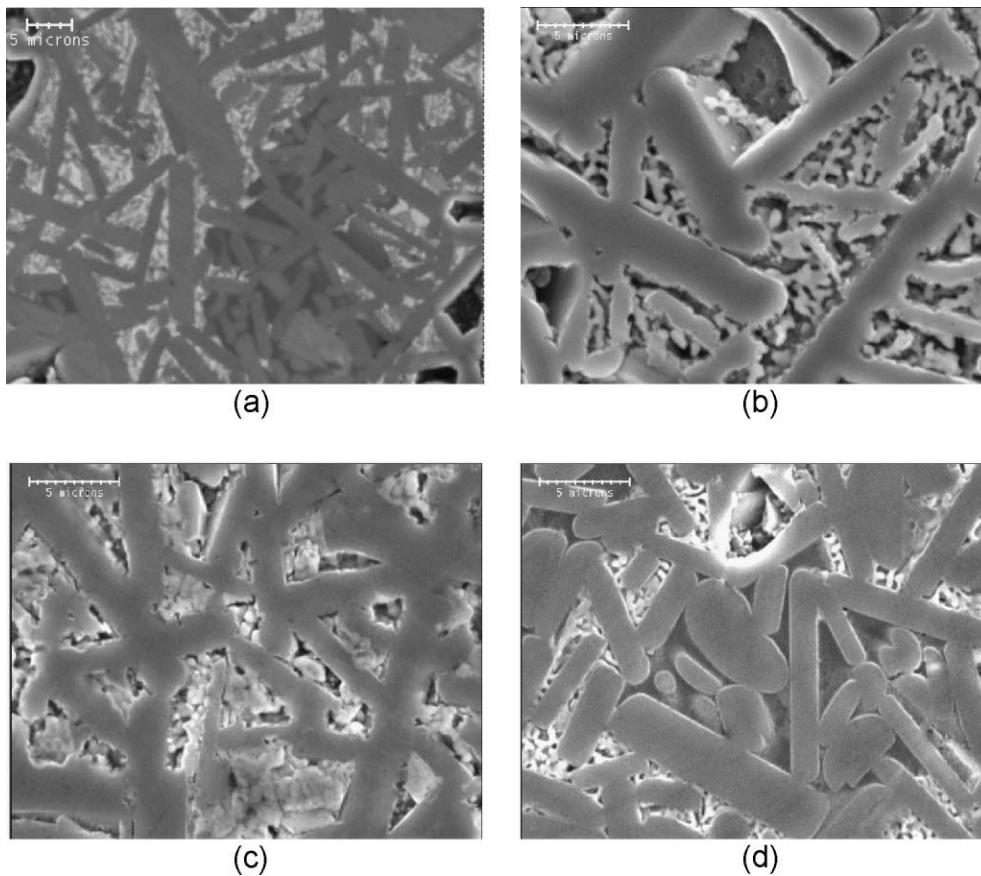


Fig. 8. Dense sintered pellets as observed by Scanning Electronic Microscopy: (a) before vaporization in an effusion cell; (b)–(d) after vaporization: (b) of the lone pellet, (c) with carborundum + alumina bed, (d) with norton + alumina bed.

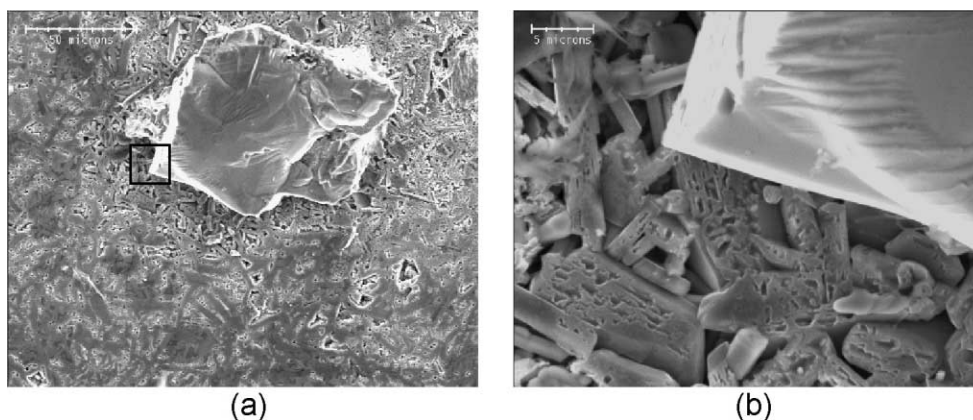


Fig. 9. Scanning Electronic Microscopy Observation: (a) a coarse SiC (carborundum) grain of the bed in close contact with the pellet, (b) magnification of the pellet grains in the contact area.

YAG, the latter being less volatile.¹¹ However the microscopic observations of pellets do not allow the determination of relative vaporization rates or weight losses.

For larger magnifications, we observe some attack or decoration (Fig. 9) of the SiC grains of the pellet heated in the Carborundum bed, especially for the grains at the surface and in contact or in the vicinity of the SiC grains of the bed. As for this pellet the weight loss is minimum, we believe that the pellet's SiC grains are more probably decorated by carbon deposits as already proposed via reaction (2) from mass spectrometric observations of the vapor pressures.

4. Conclusion

The mass spectrometric observations of Al(g), Al₂O(g), SiO(g) and CO(g) vapor pressures over the pellets—already sintered—and over the powder beds—carborundum or norton SiC—showed that the vaporization kinetics are different, the vaporization kinetics of the pellet being always lower whatever is the gaseous species observed, except for CO(g) in the norton bed.

Bringing the two materials—pellet and bed—together will inevitably lead to matter transfer that will change the total matter losses. We observed that the Al(g) and Al₂O(g) pressures were slightly disturbed, meanwhile the SiO(g) and CO(g) pressures changed drastically. For the pellet in the Carborundum bed, the CO(g) pressure increased meanwhile in the norton bed the CO(g) pressure decreased.

These variations are plus or minus counterbalanced by the SiO(g) pressure variations, although these pressures remained higher than the pressure over the lone pellet. Thus, contrarily to what is expected when using a powder bed, the alumina losses may be only slightly counterbalanced, and the major effect of the powder bed resides in the modification of the CO/SiO pressure

ratio which is imposed during the sintering process. This ratio is significant not only of the oxygen potential mean value but also of the carbon activity as discussed previously.¹¹

The nature of the SiC powder used in the beds changes drastically the above ratio and we observed carbon deposits at the surface of the sintered pellet when using a bed with high CO/SiO ratio, i.e. the carborundum one. For this bed, the sintered pellet has a minimum weight loss^{4,5} meanwhile the bed is really an alumina buffer with an important weight loss, not only toward the pellet but also outside by effusion. Using the norton SiC bed, the pellet losses are more important and we did not observe carbon precipitates. These observations are directly comparable with those of the sintering process at higher temperatures, probably because the effusion cell is a acceptable chemical reactor when compared to the sintering containers and the main chemical reactions remain the same over a large temperature range.

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