

Direct measurement of drainage curves in infiltration of SiC particle preforms: influence of interfacial reactivity

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Received: 18 January 2008 / Accepted: 21 April 2008 / Published online: 13 May 2008
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Abstract We present dynamic measurement of drainage curves in two systems having relevance to metal matrix composite processing, namely SiC/Al and SiC/Al-12.2at%Si. Data show that liquid/solid chemical reactions that cause a lowering of the contact angle do indeed drive spontaneous ingress of metal into the preforms at fixed applied pressure; however, these also hinder infiltration under continuous infiltration, lower pressurization rates causing a reduced level of penetration by the metal at given pressure. Metal/reinforcement chemical interactions that can drive wetting by lowering the contact angle are, therefore, not necessarily beneficial in the pressure infiltration processing of particle reinforced metals.

Introduction

It is well known from sessile drop data that wetting in reactive systems is often time-dependent. The underlying reason is that chemical interaction alters the phases and chemical species in presence at the triple line of mutual contact between the melt, the solid, and the atmosphere. As a result the triple line moves under the action of changing capillary forces, at a rate that can be governed either by

local reaction kinetics or by transport of reacting species through the liquid or vapor phases [1–3].

One of the most practically important wetting systems in metal matrix composite material processing is that of solid silicon carbide wetted by aluminum-based melts. This system has been extensively characterized: there are several articles in the literature where sessile drops are used to characterize wetting and its links with reactivity [3–8] for both the Al/SiC and Al-Si/SiC systems. It is known that the presence of silicon in aluminum does not significantly modify the wettability of SiC [3, 5, 8]. It also renders Al_4C_3 unstable beyond roughly 11.3at%Si at 750 °C, thus preventing carbide formation [9]; however, formation of this compound being slow this generally does not influence the wetting of SiC [3–5].

A salient feature of SiC is that below a temperature range that depends on the atmosphere and lies near 1100 °C in high vacuum, the outer surface of the carbide is constituted of a nanometric layer of SiO_x ($x \leq 2$) or silicon oxycarbide; above this temperature the SiC surface is graphitized [3–5]. Below this temperature, therefore, wetting of SiC is initially akin to wetting of silica and features a high initial contact angle (θ_1 in the terminology of Ref. [5]), near $110 \pm 10^\circ$ for aluminum, both in high vacuum [3–5] or neutral atmospheres [10]. Thereafter, sessile drops spread on the SiC surface, to gradually attain a lower angle (θ_2 in the terminology of Ref. [5]), either characteristic of wetting of SiC by the melt and scattered in the range $60 \pm 10^\circ$ (depending in particular on the activity of oxygen in the system) [5] or, if alumina formed by reduction of silica coats the SiC surface, a value near 90° characteristic of aluminum on alumina [4]. The rate of gradual decrease from θ_1 to θ_2 is governed by the rate of reduction of the silica layer, to form either alumina plus silicon in solution within the melt or, under high vacuum, the volatile species Al_2O and SiO, which leave the triple line through the

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atmosphere [5]. Thereafter, the contact angle remains nearly constant.

Depending on the experiment, particularly at lower temperatures and higher oxygen activity in the atmosphere, these stages can be preceded by one of much poorer apparent wetting. This stage is caused by the presence of a film of alumina on the aluminum-based drop [3–5]. In high vacuum this alumina layer evaporates. Data from pressure infiltration experiments show that it also does not interfere with wetting in forced infiltration [11–13]; although there may be oxygen species along the liquid surface in pressure infiltration, data thus show that any thick alumina film at the liquid surface is dynamically skimmed off from the moving molten metal front by the solid preform material, and hence does not interfere with wetting. In pressure-driven infiltration, therefore, this first stage of poor wetting has no importance.

In Ref. [11] we present a study of the wetting of silicon carbide at 750 °C by 99.99% pure aluminum and by the aluminum-silicon eutectic (Al-12at%Si) using a method that differs from the sessile drop experiment. This alternative method is a highly instrumented version of pressure infiltration that measures wetting by means of “drainage” curves. These curves plot, for the system at hand, the capillary pressure that must be applied on the non-wetting metal for it to fill a given fraction of open pore space within a porous preform of the solid phase [14]. What is found for the SiC/Al-Si system is that data are fully consistent with sessile drop data for the wetting of unreacted SiO₂ by oxide-free aluminum at that temperature [5]. This makes sense since (i) SiO₂ typically covers SiC and (ii) in these experiments infiltration is forced; as a result the rate of triple line motion is well above the “natural” rate that would be dictated by reaction kinetics. Therefore, the infiltration and the reaction processes are decoupled in time, full coverage of the solid by the liquid occurring well before significant interfacial reaction has taken place.

The Al/SiC and Al-12Si/SiC systems are, however, prone to two possible interfacial reactions, as detailed in what precedes. One would therefore expect that dynamic wetting effects be observable also in infiltration experiments when the rate of infiltration is sufficiently low for interfacial reaction to take place concomitantly with wetting. In this regime, of rapid reaction and lower rates of infiltration, interaction between the two phenomena is expected. This interaction can, depending on the system, take a variety of forms, reaction in some instances driving metal infiltration (examples are listed in Refs. [15, 16]), or causing in other instances blockage of infiltration if solid reaction products close the pores and “choke” infiltration prematurely, e.g. Refs. [17, 18].

We present here an exploration of the interplay between reaction and wetting during the infiltration of SiC by Al

and Al-12%Si using the same experimental approach as in Ref. [11] but conducting experiments under conditions of slower or interrupted pressurization. The results show that chemical reactions that drive wetting in the sessile drop configuration can, in the same system, either aid or hinder wetting during pressure infiltration, depending on conditions.

Experimental procedures

The infiltration equipment and the setup for the direct measurement of drainage curves are presented in Refs. [11, 14, 19]. This apparatus enables the study of kinetic effects in reactive infiltration since the saturation is tracked dynamically.

In preliminary experiments it was found that dynamic effects are most evident with the finest (F1000) SiC particles; this of course is as expected, given the higher specific surface and smaller diffusion distances. Such particles were thus used, namely black quality SiC F1000 powder with purity >98%, and 6.5 μm of average diameter provided by ESK-SiC-GmbH, Frechen, Germany, identical to those used in Ref. [11]. The Al and Al-12.2at%Si melts were also the same as in Ref. [11].

The SiC particles were prepared following the same procedure as was used for all infiltrated powder preforms in Ref. [11], namely stirring in HCl:distilled water = 1:1 for 30 min, washing with distilled water, stirring in ethanol for 5 min following by sedimentation and removing the ethanol (repeated three times), and finally drying at 353 K (80 °C). The SiC particle surface was thus covered with its native oxide cleaned for metallic impurities by this procedure. XPS analysis of the thus cleaned SiC particle surface yielded the following surface compositions: 23 wt% O, 23 wt% C, 54 wt% Si (see Table 1 of Ref. [11]).

To evidence reaction-related effects in capillarity during infiltration, two experiments were conducted:

- (1) experiments similar to those in Ref. [11], albeit using a lower constant rate of pressurization, namely 0.05 MPa/s (versus 0.13 MPa/s in Ref. [11]); and
- (2) pressurization was interrupted after a certain amount of metal ingress into the preform at 0.13 MPa/s, so as to detect and monitor spontaneous reaction-driven ingress of metal into the preform.

Results

Microstructure of the composites

The microstructures of SiC F1000 infiltrated at 750 °C to a maximum pressure of 10 MPa with Al and Al-12.2at%Si

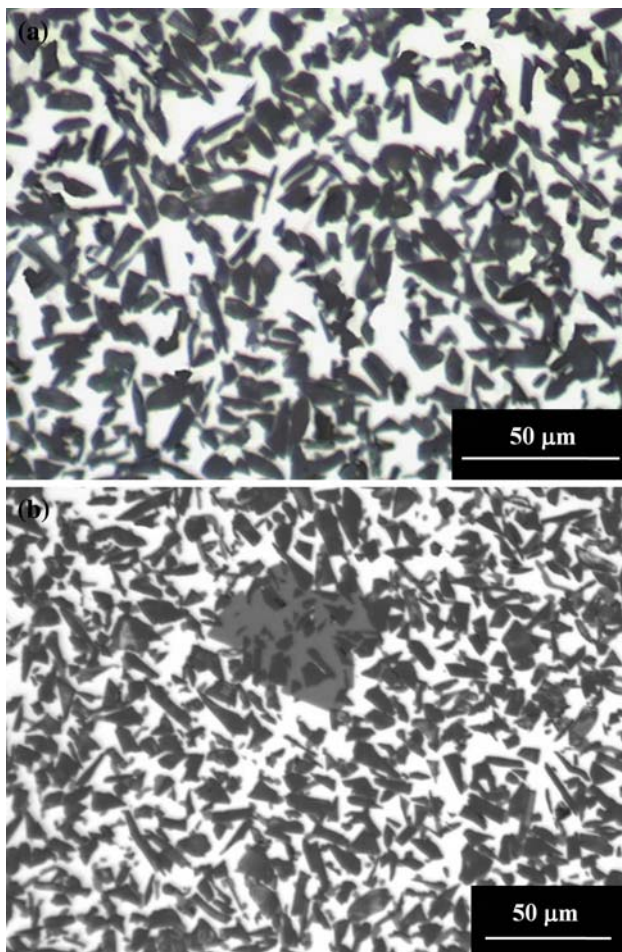


Fig. 1 Optical micrographs of SiC F1000/Al (a) and SiC F1000/Al-12.2at%Si (b), infiltrated at 750 °C and 10 MPa. The pressurization rate was 0.13 MPa/s

are shown in Fig. 1a and b, respectively, for the higher pressurization rate of 0.13 MPa/s, and in Fig. 2a and b, respectively, for the lower pressurization rate of 0.05 MPa/s. At the higher pressurization rate, Fig. 1, full infiltration is achieved, the matrix being free of porosity (see also Figs. 1 and 2 of Ref. [11]). At the lower rate of infiltration, Fig. 2, one notices the presence of dark regions corresponding to residual matrix porosity. These pores, bigger and more frequent with pure aluminum than with Al-12.2at%Si, have a characteristic length scale of several particle diameters. Figures 1b and 2b also show, in darker gray color, Si crystals growing in the matrix, typical of microstructures in solidified eutectic Al-Si alloys.

Drainage curves

Influence of reduced pressurization rate

Experiments with SiC F1000/Hg at three different pressurization rates were performed (the viscosity of liquid

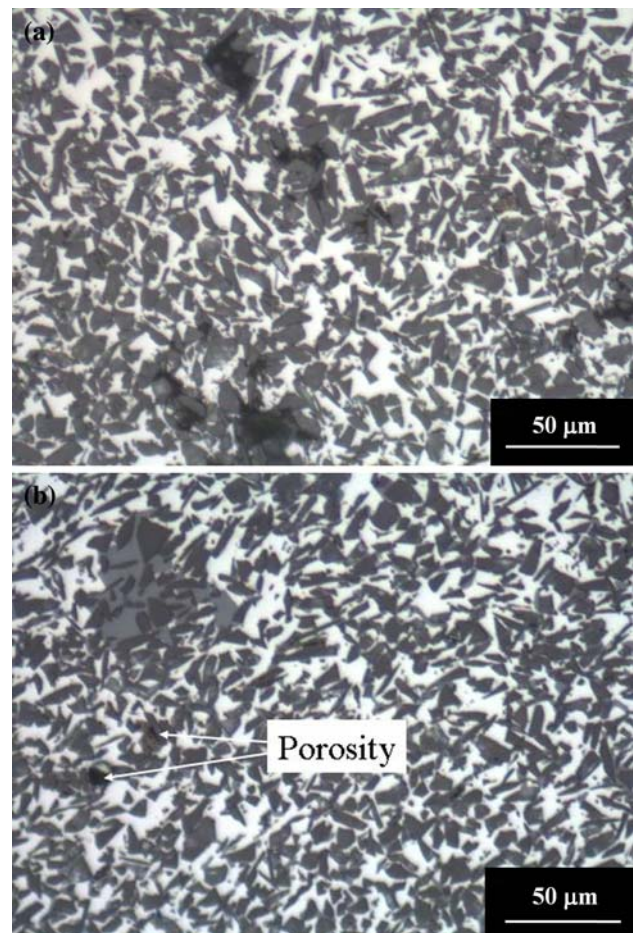


Fig. 2 Optical micrographs of SiC F1000/Al (a) and SiC F1000/Al-12.2at%Si (b), infiltrated at 750 °C and 10 MPa. The pressurization rate was 0.05 MPa/s

mercury at room temperature and liquid aluminum near its melting point are within a factor of two of one another [20]). The three curves superimpose, Fig. 3, showing that (i) the experiment features good reproducibility and (ii) there is no intrinsic influence of pressurization rate in the absence of kinetic effects in wetting. Furthermore, all drainage curves measured with mercury obey the Brooks and Corey correlation, displaying the same pore size distribution index λ as curves collected for the same SiC particles with Al or Al-12at%Si at the higher pressurization rates used in Ref. [11]. This, together with the consistency of data in Ref. [11] with wetting of unreacted SiO₂ by the two melts used in this work, shows that the 0.13 MPa/s data are characteristic of wetting in this system free of the influence of interfacial reactions.

The drainage curves measured upon infiltration of SiC F1000 with Al and Al-12at%Si at the reduced pressurization rate of 0.05 MPa/s are given in Fig. 4 together with the data measured for the same systems at 0.13 MPa/s. It is seen that the shape of the drainage curves is different at the lower pressurization rate. When plotted in the

appropriate coordinates, the curves measured at 0.05 MPa/s also depart from the Brooks and Corey correlation that is found to be obeyed by both these systems at the higher pressurization rate and mercury. Furthermore, it is found that, in infiltration with pure aluminum, at the lower pressurization rate full saturation is not attained; rather, the final metal saturation (percent pore space filled with metal) reached a plateau value that densitometric measurements confirmed to be 87%. This agrees with the observed presence of porosity in the composite, Fig. 2a. With the Al-12at%Si melt nearly full saturation is eventually reached, but whereas the curve gathered at 0.13 MPa/s reaches full saturation at 4 MPa, that measured at 0.05 MPa/s does not reach a saturation near unity before 10 MPa. Metallography indeed shows the presence of only minor porosity at 10 MPa in this system, Fig. 2b. Results on both systems are in contrast with data in Ref. [11], where with a rate of 0.13 MPa/s a pore-free matrix results at 10 MPa in this system, Fig. 1.

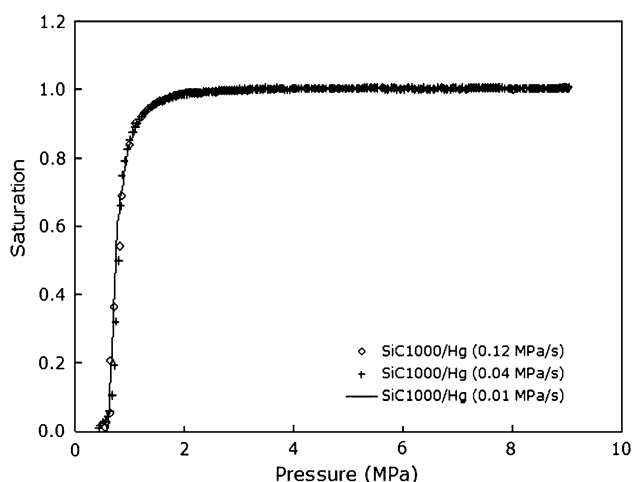
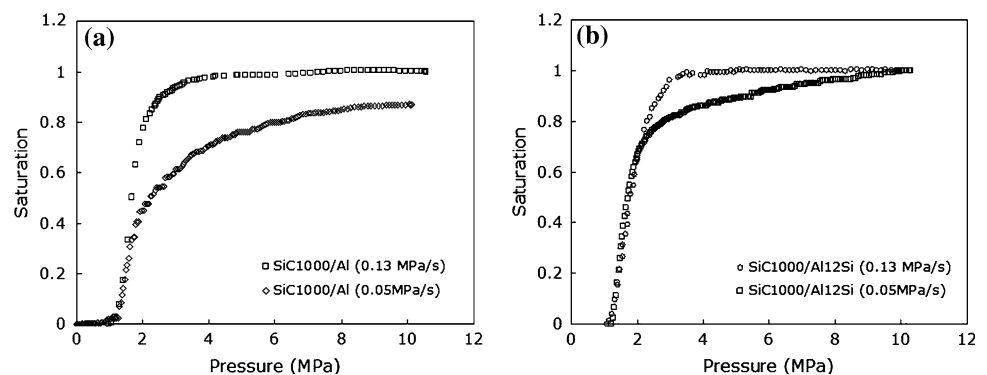


Fig. 3 Drainage curves of SiC F1000/Hg at room temperature and in three pressurization rates of 0.01, 0.04, and 0.12 MPa/s

Fig. 4 Comparison of the drainage curves at 750 °C and 10 MPa of (a) SiC F1000/Al and (b) SiC F1000/Al-12.2at%Si, measured at the two pressurization rates of 0.13 and 0.05 MPa/s



Interrupted pressurization

The preceding data show that, in both systems, interfacial reaction represents a hindrance during pressure infiltration: for a given pressure, slower infiltration and, hence, more advanced interfacial reaction causes a lowering of the fraction pore space filled with metal. This result is a priori unexpected, since over time, and hence with increasing interfacial chemical interaction, reaction causes a lowering of the contact angle and hence should aid wetting and drive infiltration.

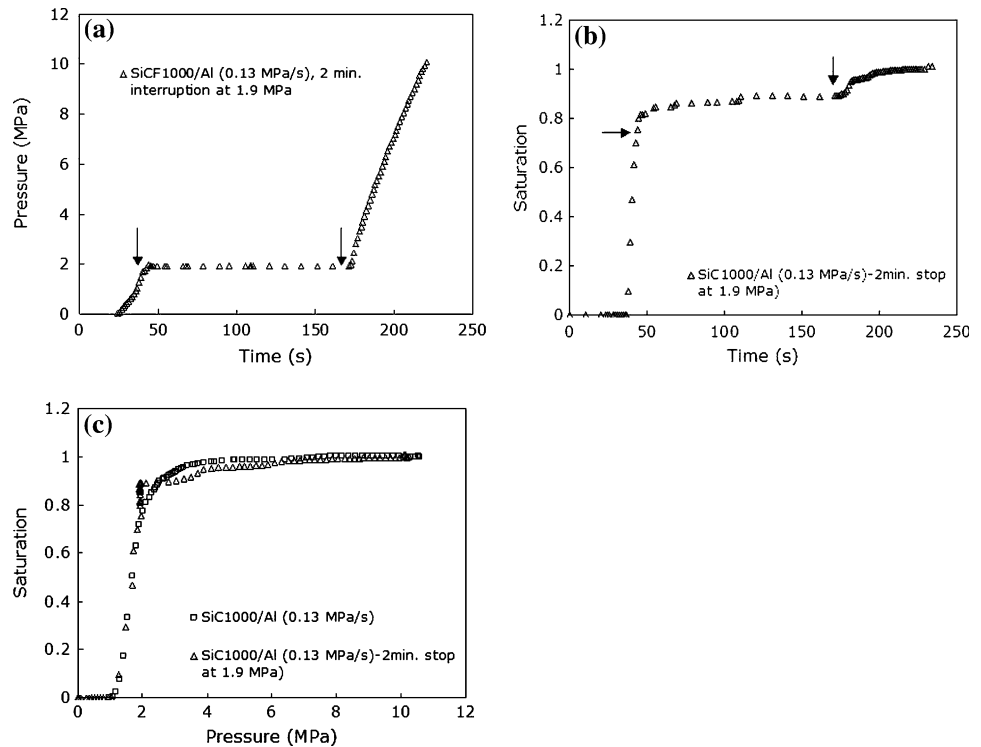
We therefore investigated whether in the absence of a finite rate of pressurization kinetic effects resulting from interfacial reactivity might drive infiltration. To this end, an experiment was started as in Ref. [11] with SiC F1000/Al using a pressurization rate of 0.13 MPa/s, but pressurization was halted at 1.9 MPa, the pressure was held constant for 2 min, and then pressurization was reinstated at the same rate of 0.13 MPa/s; the imposed pressure cycle is given in Fig. 5a. The progressive increase of saturation with time is plotted in Fig. 5b, and in Fig. 5c the saturation/pressure curve resulting from this experiment is superimposed on the 0.13 MPa/s drainage curve measured for this system.

The aim of this experiment was to track any spontaneous ingress of metal. It is seen that the saturation indeed gradually increases, spontaneously, from a saturation of 0.78 to about 0.9 at the fixed pressure of 1.9 MPa, the rate of infiltration decreasing gradually with time. After pressurization is reinstated, the drainage curve first falls below, and then gradually rejoins, the curve obtained under steady pressurization, which is also plotted in Fig. 5c.

Discussion

The present experiments show that, when the imposed rate of pressurization of the metal is sufficiently low, kinetic effects influence capillarity in infiltration. Furthermore, the

Fig. 5 Pressure–time (a), saturation–time (b), and saturation–pressure (c) curves for SiC F1000/Al at 750 °C, for an experiment conducted up to a pressure of 10 MPa, at $dP/dt = 0.13$ MPa/s with a 2-min interruption at 1.9 MPa (the arrows in (a) and (b) show the interval of interruption). Plot (c) also gives the drainage curve measured for this system under steady pressurization at $dP/dt = 0.13$ MPa/s



data show that these effects can, in the same system, either impair penetration of the preform (when the pressurization rate is low, at 0.05 MPa/s in this work) or drive infiltration (when pressurization is stopped).

Before attempting to explain the observed effects, a few remarks about the present experiments are in order.

- (1) In both Ref. [11] and the experiments presented here, the pressurization rate was kept sufficiently low so as to maintain viscous pressure losses well below capillary pressures; relevant calculations are given in Ref. [21] and experimental evidence for an absence of significant viscous loss in this regime of infiltration is provided by Fig. 3 for mercury intrusion at three different infiltration rates. Kinetic effects observed here are therefore entirely capillary in origin.
- (2) Since silicon in molten aluminum exerts a negligible influence on wetting (as known from sessile drop data in the literature [3, 5, 8] and confirmed by the data in Ref. [11]), silicon concentration gradients, which might arise in the present infiltration experiments due to interfacial reaction coupled with fluid flow, should be inconsequential. This is a significant simplification of the present system.
- (3) A complication, on the other hand, is that the atmosphere influences the kinetics of wetting in this system. Indeed, reported rates of motion of the triple line in sessile drop experiments are for ultrahigh vacuum (total pressure $P \leq 5 \times 10^{-5}$ Pa and oxygen

partial pressure of about 10^{-15} Pa [4, 5]), where the process is governed by the rate of evaporation of volatile oxides (SiO, Al₂O) [3–5]. In the present work, although the oxygen partial pressure measured inside the chamber is very low (10^{-17} Pa), the total residual gas pressure is not better than 0.2 mbar (it is generated by a mechanical pump, and kept by the molten metal sealing the preform from the surrounding atmosphere) [14]. Also, the temperature is somewhat lower, at 750 °C. For these reasons the de-oxidation process is probably not as rapid as in ultrahigh vacuum, such that spontaneous, reaction-limited, triple line velocities in the present system are likely lower than in ultrahigh vacuum sessile drop experiments. Values of triple line velocities taken from the curves in Ref. [4] are near 7×10^{-2} μm/s for Al on oxidized SiC at 900 °C and also for Al-19.5at%Si on SiC at 800 °C, and about 4×10^{-2} μm/s for Al on SiC at 800 °C. We thus take 10^{-1} to 10^{-2} μm/s as an order of magnitude estimate of the maximum possible value of spontaneous triple line velocities of Al and Al-Si alloys over SiC in our experiments.

- (4) In the present technique, we measure not the rate of motion of the triple line, but the rate of increase of saturation, i.e., the rate at which the metal volume fraction present within the porous preform increases. The link between this quantity and the triple line velocity depends on the precise infiltration path followed by the metal as it fills open pores within

the preform. Given the intricate geometry of the ceramic particles, and also the complex morphology of pores found in partially infiltrated samples (see Figs. 1 and 2 of Ref. [19] where this is clearly visible on an analogous but non-reactive system), this infiltration path, and hence the trajectory of the triple line, is very complex.

A lower limit of the total length of this trajectory is obviously the particle radius, of about $3\ \mu\text{m}$ (Table 1 of Ref. [11])—the corresponding infiltration flow path is, then, one of metal gradually filling all menisci between contacting particles in the last phases of infiltration. The time to infiltrate, at constant pressure and with a spontaneous triple line velocity of 10^{-1} to $10^{-2}\ \mu\text{m/s}$, the preform is then on the order of 30–300 s. As seen in Fig. 5, in the present interrupted experiment, the saturation increases from 80 to 90% in 120 s, which is not out of line with this crude estimate.

This estimate suffers, however, from two limitations. First, triple line velocities characteristic of sessile drop experiments in high vacuum were used; it is, as mentioned earlier, likely that *lower* velocities than 10^{-1} to $10^{-2}\ \mu\text{m/s}$ are obtained in the present experimental conditions since lower rates of evaporation of volatile oxides are expected. Secondly, the infiltration path is clearly more complex than was assumed in this simple calculation; in particular, its length is very probably longer than one particle radius since pores present in the later stages of infiltration span several particles (Figs. 1 and 2 of Ref. [19]): the triple line is correspondingly likely to travel distances several times the pore diameter to close off the last pores. To increase the saturation by 10% in 120 s as observed here, spontaneous triple line rates of motion must therefore have been, on average, significantly *higher* than 10^{-1} to $10^{-2}\ \mu\text{m/s}$.

An assumption that might reconcile these two contradictory conclusions is that the line moves by jumps during spontaneous infiltration. In this scheme, spontaneous spreading serves to overcome local capillary barriers, the metal moving much more rapidly past such localized pinning points under the joint action of capillary forces and the applied pressure. The existence of such jumps, called “Haines jumps,” is indeed a well-known feature of capillarity in porous media [22]. With this interpretation, the steadily decreasing rate of spontaneous infiltration at fixed pressure, Fig. 5b, may be explained by the gradual exhaustion of pinning points that can be overcome by reaction-driven triple line motion. We note that, given the observation made in Ref. [11] that the total work spent to force the metal into the preform equals, with relatively good precision, the total thermodynamic work of

immersion, these jumps must be accompanied by comparatively little expenditure of irreversible energy, be it capillary or kinetic.

Turning to the influence of pressurization rate, we first note that the total time for infiltration of the preform is, at 0.13 MPa/s, around 70 s. This is on the order of the time estimated for the shortest possible triple line trajectory using upper-bound estimates of the spontaneous triple line velocity. It thus is very likely that the average triple line velocity was, during pressure-infiltration at 0.13 MPa/s, well above the spontaneous reaction-driven triple line velocity. This conclusion in turn agrees with the observation that wetting in infiltration at this rate of pressurization is typical of non-reactive wetting of silica by aluminum, as shown in Ref. [11].

Lowering the pressurization rate from $dP/dt = 0.13\ \text{MPa/s}$ to $dP/dt = 0.05\ \text{MPa/s}$ causes a downward shift in the drainage curves, Fig. 4. Thus, while it can cause spontaneous ingress of the metal into the preform, reaction in fact impairs infiltration during pressure infiltration. One possibility is that this effect may result from a change in the kinetics of interaction between wetting and any oxygen in the surrounding gas near the wetting front; such effects are for example found in wetting of alumina by aluminum (see for example [23, 24] and references therein). One would, then, however expect that drainage curves show an equally strong transition in drainage curves with temperature, as temperature too will affect the kinetics of interaction with oxygen. This is not seen, data in all our experiments to date with the present apparatus agreeing with expectations from sessile drop data for conditions where no interference exists between liquid metal surface oxidation and wetting, as indicated in the “Introduction” section.

An alternative explanation for the effect of pressurization rate is that reaction may cause topological changes in the pore shape, narrowing these at “bottlenecks” that govern the flow path under the influence of capillary forces. In the SiC/Al system, reaction will first cause aluminum oxide formation by reduction of the silica layer under conditions of slow volatile oxide evaporation, and given sufficient time also platelets of aluminum carbide that extend into the matrix with pure Al (see Figs. 1 and 2 of Ref. [11]). Both reaction products are not very well wetted by aluminum [3] and, if their shape is complex, their presence will create geometric capillary barriers to infiltration. If reaction is sufficiently extensive, the reaction products may also lower the permeability of infiltrated portions of the preform, causing viscous pressure losses to become noticeable or even choking of metal flow by pore closure.

That the influence of reduced pressurization rate be attributed to the physical presence of reaction products,

rather than a relation between infiltration rate and intrinsic capillary parameters (the wetting angle and work of immersion), is consistent with data in that (i) in SiC F1000/Al-12.2at%Si, where reaction is less pronounced than in SiC F1000/Al (forming only aluminum oxide by reduction of the native oxide layer but not aluminum carbide), the effect be less marked, and (ii) there be a “memory” effect after the 120 s hold at 2 MPa, such that after spontaneous ingress of the metal at fixed pressure, upon further pressurization at 0.13 MPa/s, the rate and extent of metal penetration be less than with similar but uninterrupted pressurization, Fig. 5b.

Conclusions

- We show that under forced, pressure-driven infiltration, interfacial reactions that cause a gradual lowering of the wetting angle in sessile drop experiments and thus aid wetting, can either aid or hinder infiltration of SiC preforms by aluminum-based melts. Specifically, while interfacial reactions can cause spontaneous penetration of partially infiltrated metal into the preform at fixed applied pressure, during infiltration driven by a rising applied pressure with negligible viscous pressure losses, interfacial reactions can hinder infiltration.
- It is proposed that these effects are due to the fact that chemical interactions cause, in addition to a lowering of the wetting angle, morphological changes at the solid/liquid interface, which can hinder wetting and infiltration.
- A corollary is that, from a practical point of view, rapid pressure infiltration is in fact preferable in processing metal matrix composites featuring interfacial reactivity, even when liquid/solid chemical interactions improve wetting in sessile drop experiments.

Acknowledgements The authors gratefully acknowledge financial support of this research from NEDO International Joint Research Grant Program, Project 01MB7, CTI project no. 6752-2 and the internal funds from the Laboratory of Mechanical Metallurgy at EPFL.

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