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High-temperature wetting and interfacial interaction between liquid Al and TiB_2 ceramic

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Abstract The wetting behaviour and interfacial interactions between liquid Al and TiB_2 ceramic have been studied by the sessile drop technique in the temperature range from 700 to 1400 °C. At about 800 °C, liquid Al starts to wet TiB_2 and at about 1000 °C it completely spreads over the ceramic. Al_3Ti and Al_2O_3 are found to be the main phases precipitating at the interface. Starting from 1000 °C, liquid Al either fill pores or penetrates along the grain boundaries of the TiB_2 ceramic. Scanning electron microscopy analysis of the interfaces evidences that the TiB_2 grains remain intact after the aluminium melt/ceramic interaction even at 1400 °C.

Introduction

Diboride compounds of the group IV transition metals (TiB_2, ZrB_2, and HfB_2) are characterised by very high melting temperatures, superior hardness, and chemical inertness to corrosive substances [1, 2] and are, therefore, very important for applications under extreme conditions. In particular, TiB_2 with a melting temperature of about 3200 °C [3] and a theoretical density of 4.52 g/cm^3 [1] is interesting as an electrode material in the aluminium industry, as temperature or wear protective coating or as constituent of light-weight composites for aerospace, automotive and defence applications [4–6]. Besides, titanium diboride is used for the microstructure refinement of cast Al-based alloys [7–9]. In general, processing and properties of these materials are mainly determined by the following features: (i) stability of TiB_2 in contact with a liquid metal or alloy; (ii) wetting, spreading and infiltration behaviour of a metallic melt in contact with TiB_2 ceramic; (iii) new phases which may be formed at the melt/TiB_2 interface.

High-temperature interaction in the Al–TiB_2 system has been investigated in various works so far (e.g. see Refs. [10–16]). Although a good wetting of TiB_2 by liquid aluminium has been generally reported, the contact angle values as well as the temperatures are rather scattered. For example, Rhee [10] studied the wetting of dense TiB_2 (98.5 % of the theoretical density) by liquid aluminium under high vacuum (better than 2.7×10^{-7} mbar) by the sessile drop technique. After keeping the system for at least 30 min at a given temperature, the equilibrium contact angle reached about 90° at 710 °C and about 60° at 840 °C [10]. Samsonov et al. [11] did not observe any wetting of TiB_2 substrates (about 95 % of the theoretical density) by liquid aluminium at 900–1000 °C in vacuum, whereas the contact angle decreased to about 75° and to about 30° after 20 min of isothermal heating at 1150 °C and at 1250 °C, respectively. Weirauch et al. studied the wetting behaviour of liquid Al on different TiB_2 substrates at a constant temperature of 1025 °C [12]. It was reported that the initial contact angle of about 120°–140° for liquid Al on a polished TiB_2 substrate (99.8 % or better purity, 99.7 % of the

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theoretical density) decreased to 0° after prolonged holding for 17 h.

Infiltration of liquid Al into TiB_2 ceramic materials and its reaction with impurities or additives have been studied in dependence on contact time for the temperature between 960 and 980 °C [13–16]. It has been reported in [13] that at 970 °C porous TiB_2 (90 % theoretical density) is rather fast penetrated by liquid aluminium (>1 mm/day), while dense TiB_2 (96 % density) is penetrated at much slower rate (>0.1 mm/day). Depending on the quality of TiB_2 or TiB_2-based materials and time, liquid aluminium penetrates either pores or pore-free grain boundaries (edges as well as faces) as established in [14–16].

The aim of the present work is a systematic study of the temperature- and time-dependent phenomena at the interface between liquid Al and TiB_2 ceramic over a wide temperature range using the sessile drop method. Application of the drop dispenser technique enables essential minimisation of the effects related to oxidation of molten aluminium. Knowledge of the wettability kinetics in the liquid Al/TiB_2 ceramic at high temperatures is important for the development/optimisation of the Al-base lightweight alloys casting using TiB_2 grain refining. The high-temperature data on wetting, penetration and phase formation at the liquid Al/TiB_2 interface are also required for the development of TiB_2-reinforced Al-based composites.

Materials and methods

The substrates for the sessile drop tests were cut in form of rectangular plates (12 mm × 12 mm) with a thickness of 1–2 mm from TiB_2 (ESK Ceramics, Kempten, Germany) by electric discharge machining. The experimentally measured density of this TiB_2 ceramic was about 4.46 ± 0.02 g/cm^3 ($\sim$98.7 % of the theoretical density). A dense microstructure with a grain size of 10–20 µm is well seen in scanning electron microscope (SEM) images taken at the fracture surface of a broken TiB_2 plate, as shown in Fig. 1a. A few TiC particles found between the grains presumably originate from carbon or boron carbide commonly used for synthesis of TiB_2 ceramic [1, 17].

The TiB_2 plates were ground with a resin-bonded diamond disc and subsequently polished using different cloths and diamond suspensions (all from Struers A/S) to an average roughness of about 0.1 µm, measured by Scanning Probe Microscopy (SPM). A SEM image of the surface of a polished TiB_2 plate is shown in Fig. 1b. Before the experiments, the substrates were ultrasonically cleaned in acetone.

Aluminium samples were prepared from high-purity metal (99.999 %, ChemPur). Directly before testing, they were mechanically cleaned and washed with isopropanol in an ultrasonic bath.

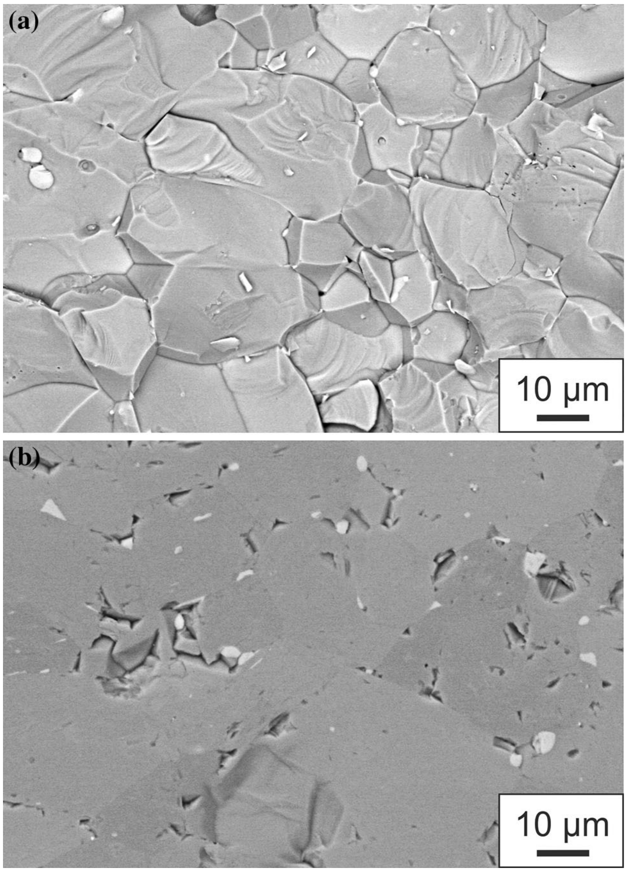

Fig. 1 SEM images of a fracture surface for a broken TiB_2 ceramic (**a**) and the surface of a polished TiB_2 plate (**b**): *grey* TiB_2 phase, *white* TiC phase

The experiments were carried out using two different procedures: (i) classical sessile drop tests, where the sample lies on the substrate and both substrate and sample are jointly heated from room temperature (so called contact heating), (ii) dispensed drop experiments, in which the sample is heated separately in a drop dispenser situated above the substrate, and a liquid drop is squeezed onto the substrate at a given temperature (non-contact heating; also called as capillary purification method).

The classical sessile drop tests with contact heating were carried out in the device described in detail in Ref. [18]. The experimental setup consisted of a horizontal resistance furnace inside a stainless steel chamber with illumination and observation windows, sample illumination and an optical system with a charge-coupled device (CCD) camera. The Al/substrate couple was placed on a boron nitride support situated in the middle of a molybdenum resistance furnace. The temperature was measured by a thermocouple located directly above the sample. The experiments were performed under a vacuum of about 1×10^{-5} mbar. The system was heated from room temperature up to 1200 °C with a rate of 10 K min^{-1}. Digital photographs of the Al/TiB_2 couples were taken in steps of 10 K during heating.

The sessile drop tests with capillary purification were performed using the experimental setup described in Ref. [19]. In this case, the Al samples were inserted into an alumina drop dispenser, which was situated above a TiB_2 substrate placed on an alumina support. This assembly was situated in the middle of a vertical tantalum resistance furnace placed in a stainless steel chamber with illumination and observation windows. The temperature was measured by three thermocouples located below and above the substrate, close to the drop dispenser. Before heating, the chamber was evacuated to about 10^{-6} Pa. At 500 °C, it was filled with high-purity argon (99.999 %). The flowing gas pressure was kept in the range of 850–900 mbar during the experiments. At a certain temperature, an Al drop was squeezed out of the dispenser onto a substrate and the sessile drop test was started. Manipulation with the substrate and drop dispenser as well as the behaviour of the Al drop on the substrate either at a constant temperature or upon heating at a rate of 10 K min^{-1} was registered by a high-speed/high-resolution CCD camera.

The digital images of the sessile drops were analysed using DROP [18] and ASTRAView [20] software. Contact angles were determined at the left and the right sides of the droplet with an accuracy of about ±2°. The relative uncertainty of the averaged contact angle was about ±5°.

The microstructure of the Al/TiB_2 interface after the high-temperature sessile drop tests was studied by scanning electron microscopy (LEO GEMINI 1530). For this, the Al/TiB_2 couples were cross-sectioned by an automatic cutting machine using a Buehler Diamond Wafering Blade and embedded into epoxy resin. The cross sections were ground to about 3 μm surface finish using successively different MD-system discs and diamond suspensions (Struers A/S). The final polishing was performed using 0.04 μm colloidal silica suspension (Buehler).

Phase analysis of the cross sections of the Al/TiB_2 couples was performed by energy-dispersive X-ray (EDX) spectroscopy and X-ray diffraction (XRD). The EDX analysis was carried out using a Bruker XFlash Detector 4010 operated at 5 keV for a better detection of boron, carbon and oxygen, as well as at 20 keV. XRD was carried out using a D3290 Panalytical X' Pert PRO diffractometer with Co K_α-radiation in a range of diffraction angles 2θ between 20° and 110°. Phases were identified using the International Centre for Diffraction Data (ICDD) database.

Experimental results and discussion

Wetting upon constant heating

Figure 2 shows the wetting behaviour for liquid Al on TiB_2 upon constant rate heating at 10 K min^{-1}. In the classical sessile drop experiment (black *rectangles* in Fig. 2), a liquid drop is observed to be formed when the temperature reaches about 780 °C, which is significantly above the melting temperature of Al (660 °C). The apparent contact angle at the Al/TiB_2 interface of about 140° remains virtually unchanged up to about 1000 °C. Then it starts to decrease slightly and exhibits a steep decrease from 135° to 12° in the temperature range between 1030 and 1200 °C. On the contrary, liquid Al squeezed out the drop dispenser at 700 °C (i.e. capillary-purified) exhibits significantly better wetting of TiB_2 (red *circles* in Fig. 2). The contact angle reaches about 85° at 750 °C and decreases to 24° upon heating to 1000 °C, followed by continuous decrease to 12° at 1100 °C.

It is known that Al has a very strong affinity to oxygen. It oxidizes very fast in the solid state at room temperature and the oxidation rate significantly increases with heating and time [21, 22]. However, upon melting and heating to about 1000 °C, the oxide layer has been reported to disappear due to the formation of gaseous Al_2O by a chemical reaction between liquid Al and solid Al_2O_3 [23]. This explains the behaviour of the Al sample in the classical sessile drop experiment. Although Al was mechanically cleaned from oxides just before inserting it into the chamber, there was enough time to form a surface oxide afterwards. The onset of wetting of TiB_2 is observed at about 1030 °C (Fig. 2) after the "self-cleaning" of the liquid Al surface. A similar effect has been reported for the liquid Al/Al_2O_3 system in Ref. [24].

As the oxidation rate of liquid Al strongly depends on the oxygen partial pressure [25], it can be assumed that the Al drop squeezed out of the drop dispenser is free of oxides at least during the first minutes of the sessile drop test in the chamber evacuated to about 10^{-6} Pa residual air pressure. This explains the essentially different wetting

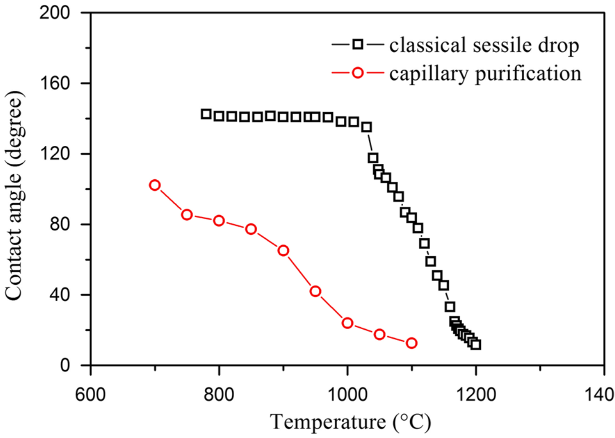

Fig. 2 Temperature dependence of the contact angle for liquid Al on a TiB_2 plate measured by the classical sessile drop (*rectangles*) and dispensed drop (*circles*) methods

behaviour for the "capillary-purified" liquid Al on TiB_2 (Fig. 2) and demonstrates the advantage of this technique. Mechanical removal of surface oxide(s) coupled with using well-purified inert atmosphere enables the wetting to start at significantly lower temperature. A similar behaviour has been reported for liquid Al on Al_2O_3 substrates [26], where initial contact angles of ~130° and of ~94° were observed in a classical sessile drop test and in a dispensed drop test at 700 °C, respectively.

Wetting upon isothermal heating

The isothermal wetting kinetics of liquid Al dispensed on the TiB_2 substrates at different temperatures is presented by the time dependences of the contact angle in Fig. 3a. Liquid Al shows rather poor wetting of the TiB_2 substrate at low temperature. For example, the average contact angle reached about 90° after 2 h of isothermal annealing at 710 °C (not shown). The wetting is slightly improved at 800 °C—the mean contact angle decreased from the initial value of $\theta_0 = 128°$ measured immediately after the drop dispensing to a final contact angle of $\theta_F = 77°$ after 2 h of isothermal holding (Fig. 3a). However, with further increasing temperature, capillary-purified aluminium exhibits a very good wetting of TiB_2 followed by a spreading over the substrate within about 20 min at 900 °C, 1 min at 1100 °C, and 20 s at 1300 °C (Fig. 3a).

Images of the TiB_2 plates with Al specimens after the isothermal holding at 800 °C and at 1000 °C are shown in Fig. 4. Starting at 1000 °C, the Al melt perfectly wets, spreads and streams down along the substrate walls and penetrates a gap between the TiB_2 plate and the Al_2O_3 support, which indicates that both ceramics are completely wetted by liquid Al at a high temperature. The cross sections of the solidified Al/TiB_2 couples after the isothermal sessile drop tests carried out at 800, 900 and 1400 °C are presented in Fig. 5. On these images, the contact angles correspond to the solidified drops and they are somewhat smaller than the contact angles measured in the sessile drop tests with liquid metal (plotted in Fig. 3a). This is due to the shrinkage of the adhered Al drop upon cooling and solidification. Figure 5b, c also show that due to a relatively large volume of Al remaining on the TiB_2 substrates the apparent contact angle is larger than zero, although Al completely wets the ceramics.

The dynamics of the Al drop spreading on the TiB_2 substrate is presented in Fig. 3b by the relative change of the drop base diameter d with time t at different experimental temperatures $(d(t) - d_0)/d_0$ (d_0 is the drop base diameter just after the drop deposition). For clarity of presentation, only the data for the first 10 min are shown. There is a change in the spreading behaviour when the temperature increases up to 1100 °C. After the initial rapid spreading within the first 10 s, the spreading rate reduces. A similar transition from the fast to a relatively slower spreading rate is observed also at higher temperatures, but on much shorter time interval, e.g. about 2 s after the drop deposition at 1300 °C. This phenomenon is supposed to be caused by pinning of the triple line due to the penetration of molten aluminium into the TiB_2 substrate at the temperature above 1000 °C, discussed below.

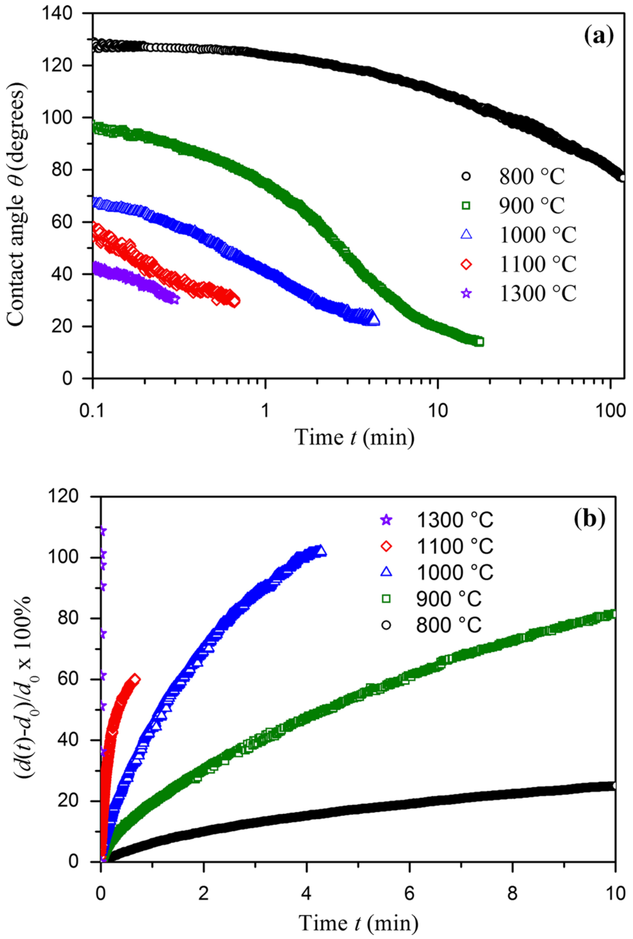

Fig. 3 Isothermal wetting kinetics for the capillary-purified liquid Al on the TiB_2 substrate at different temperatures: **a** in situ measured contact angle θ versus time; **b** relative change of the drop base diameter d with time t at different temperatures (d_0 is the drop base diameter just after the drop deposition)

It is known that TiO_2 forms rather easily on the surface of monolithic TiB_2 at room temperature [1] and it can grow upon heating due to residual oxygen in the experimental chamber [27]. In this view, it is interesting to recall the works, where the wetting behaviour of liquid Al on TiO_2 has been studied. For example, Avraham and Kaplan [28] reported the contact angle of about 150° for liquid Al on TiO_2 at 700 °C, which decreased to about 85° by heating up to 1000 °C. Sobczak et al. [29] found that the contact angle for capillary-purified molten Al on TiO_2 equals to 80° at 900 °C. In our study, liquid Al on TiB_2 substrates

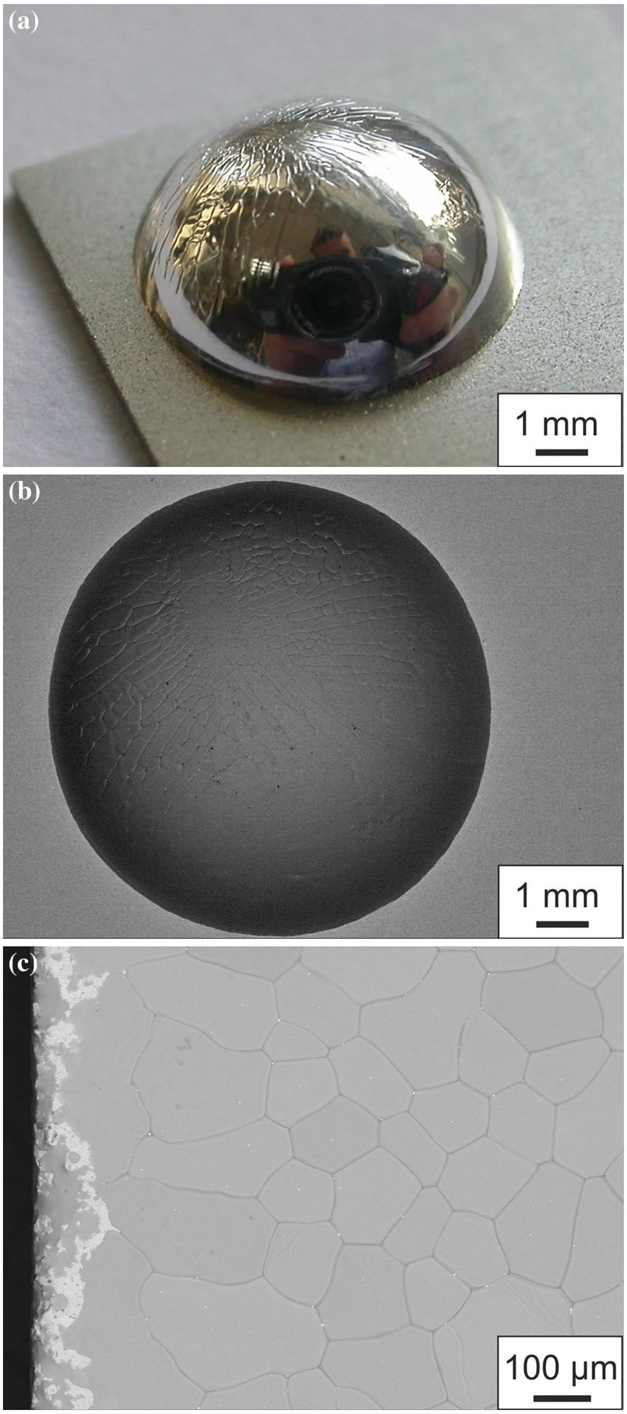

Fig. 4 Al specimens on TiB_2 substrates after isothermal sessile drop tests carried out for 2 h at 800 °C (**a**, **b**) and for 15 min at 1000 °C (**c**): **a** general view (optical microscope); **b**, **c** top view (SEM)

exhibits a better wetting (Figs. 2, 3) and the final contact angles well agree with the results reported by Rhee [10]. This suggests that either the amount of Ti oxide on the surface of TiB_2 substrate was small or it is reduced by liquid Al. The latter is supposedly to be evidenced by appearance of the Al_2O_3 and Al_xTi particles at the Al/TiB_2 interface after the sessile drop tests carried out at temperatures from 710 to 1400 °C.

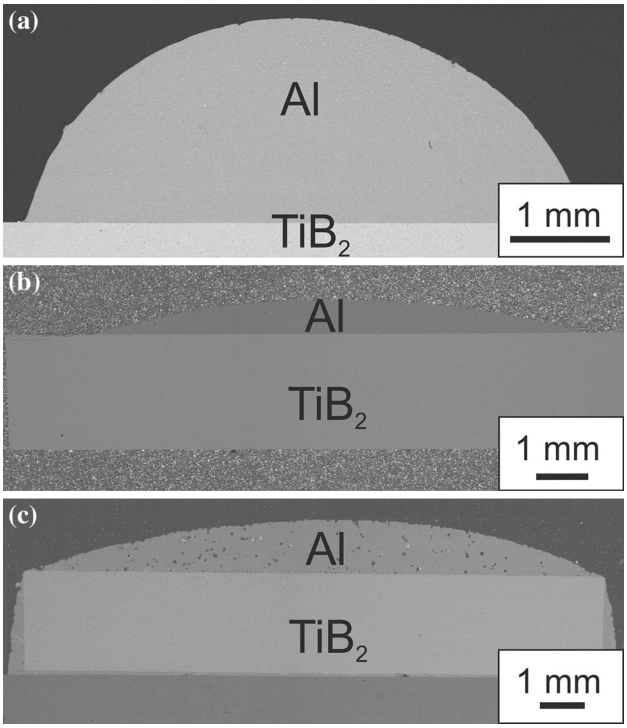

Fig. 5 SEM images of cross-sectioned Al/TiB_2 couples after isothermal sessile drop tests carried out at different temperatures for different times: **a** 800 °C, 2 h; **b** 900 °C, 1 h; **c** 1400 °C, 15 min

Penetration and interfacial reactions

Magnified SEM images of the Al/TiB_2 interface are shown in Figs. 6, 7 and 8. The interfacial area between TiB_2 substrate and Al_2O_3 support filled by liquid Al at 1000, 1300, and 1400 °C is shown in Fig. 9. The Al/TiB_2 interface is smooth and planar for all solidified samples. Aluminium and TiB_2 phases are distinctly separated in the couples kept at 710 °C, 800 °C (Fig. 6a) and 900 °C. A sharp interface is also observed for the Al/TiB_2 couples tested at 1000 °C (Fig. 6b) and even at 1400 °C (Fig. 7). However, in these cases, Al is found in the TiB_2 ceramics either in the pores, as it can be seen in Fig. 6b (1000 °C), or also at the grain boundaries, as in Fig. 7 (1400 °C). In general, these observations correlate with investigations performed at 960–980 °C [13–16] and show that the penetration rate rapidly increases with increasing temperature. The average penetration depth extends up to about 250 µm after the sessile test carried out for 15 min at 1400 °C.

TiB_2 ceramic and Al are characterised by quite different coefficients of thermal expansion: $\alpha_{TiB_2} = (6.7 - 8.0) \times 10^{-6} K^{-1}$ in the temperature range between 20 °C and 1000 °C (ESK Ceramics), while $\alpha_{Al} = (20.9 - 33.7) \times$

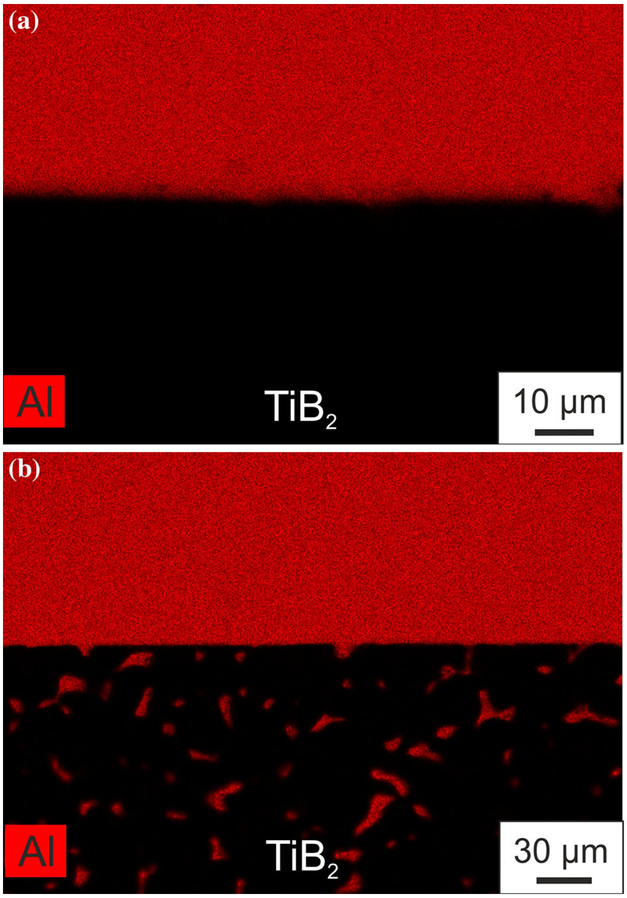

Fig. 6 SEM/EDX images of the Al/TiB_2 interface after the isothermal sessile drop tests carried out for 2 h at 800 °C (**a**) and for 15 min at 1000 °C (**b**)

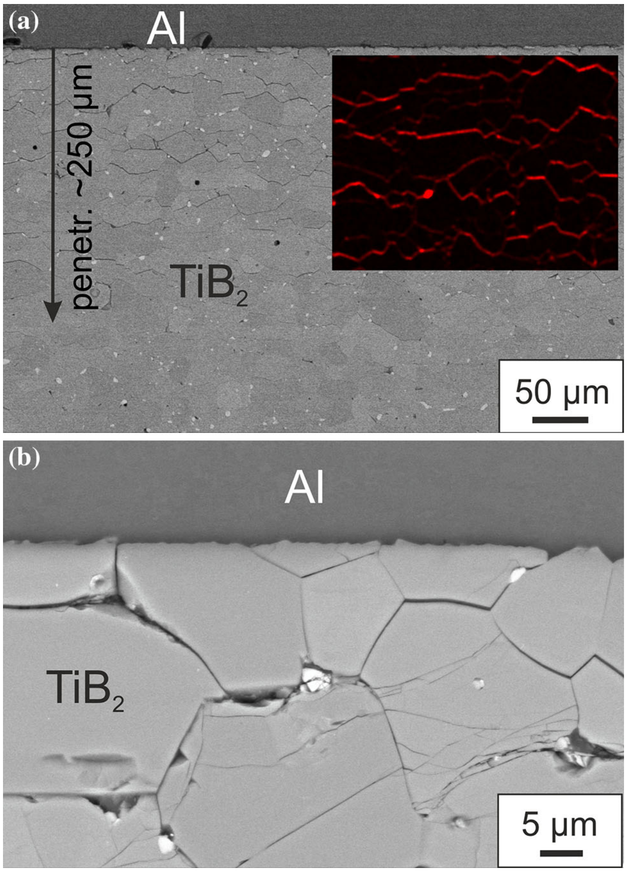

Fig. 7 Aluminium penetration along TiB_2 grain boundaries at 1400 °C: SEM images of the Al/TiB_2 interface. *Inset* in panel (**a**) shows an enlarged SEM/EDX map of Al distribution in a near-interface area. Panel (**b**) illustrates microcracks in TiB_2 grains

$10^{-6}K^{-1}$ between 20 °C and 500 °C (own measurements). Because of this, cracking of TiB_2 grains has been observed in the near-interfacial area of the substrates penetrated by liquid aluminium, as it is depicted in Fig. 7b.

As it has been mentioned, Al-rich Al_xTi particles were observed by SEM/EDX at the Al/TiB_2 interface after the sessile drop tests carried between 710 and 1300 °C. The composition of these particles is very uncertain for low temperatures, whereas after testing at 1400 °C the stoichiometric Al_3Ti phase (melting temperature $T_m = 1396$ °C [3]) with needle-like structure has been found to precipitate at the Al/TiB_2 interface and to grow into the molten aluminium (Figs. 8, 9). At the same time, SEM analysis suggests that TiB_2 grains remain intact in all experiments, in agreement with a very low solubility of TiB_2 in liquid Al [3]. Therefore, it can be supposed that the Al_xTi particles are formed due to the reaction of liquid Al with TiO_2 existing on the surface of the substrate or with TiC particles found in the TiB_2 ceramics (Fig. 1). We have also observed Al_2O_3 particles at the Al/TiB_2 interface in the samples after the tests carried out at 710–1400 °C, as it can be seen in Fig. 8 for the temperature of 1400 °C. This is in line with the reports on high-temperature interactions between liquid Al and TiO_2 [28–33] and formation of Al_2O_3 particles and Ti dissolved in Al by the reduction reaction:

$$4Al + 3TiO_2 = 2Al_2O_3 + 3Ti. \quad (1)$$

Hence, the reaction mechanism in our study can be described as follows. Liquid Al placed on TiB_2 substrate initially interacts with TiO_2 surface oxide. Al_2O_3 crystals nucleate at the drop/substrate interface and grow inside the drop, whereas pure Ti is released into the molten Al, similar to that observed in the Al/TiO_2 couples at 900, 1000 and 1100 °C in work [29]. Upon cooling, Al_3Ti phase precipitates at the Al/TiB_2 interface.

The very few Al_4C_3 and $(Al,Ti)_xC$ particles detected at the Al/TiB_2 interface after the sessile drop tests carried out at 710–1400 °C (not shown) as well as the very few Al_xTi particles found in the pores of the TiB_2 substrates are supposed to be formed due to reaction of molten Al with TiC impurities. This is in line with the Al–Ti–C phase

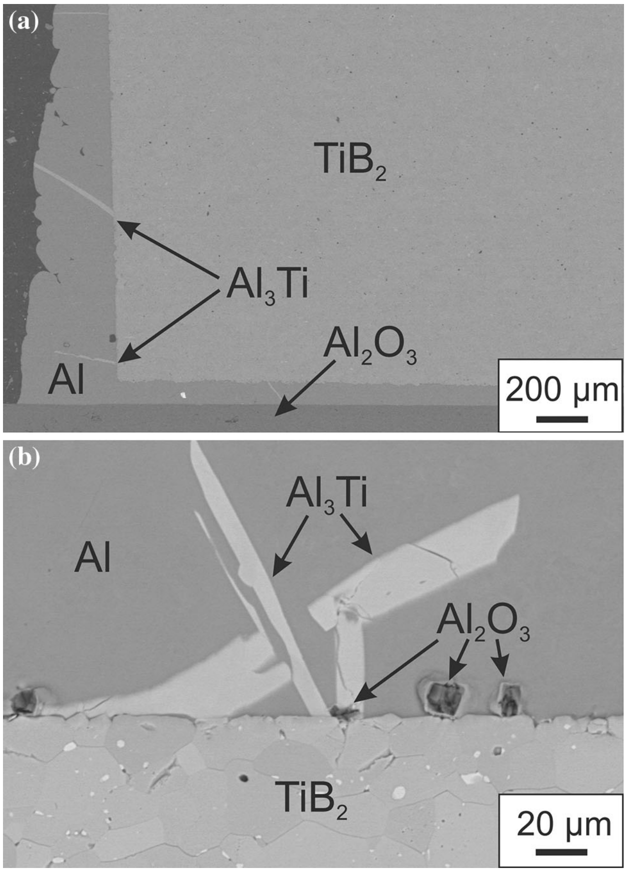

Fig. 8 SEM images of the Al/TiB$_2$ interface after the isothermal sessile drop tests carried out for 15 min at 1400 °C. Al$_3$Ti and Al$_2$O$_3$ phases are marked

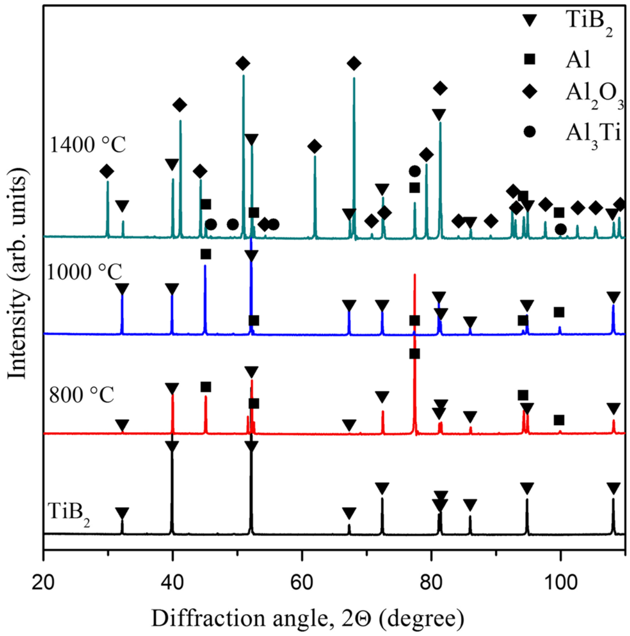

Fig. 9 XRD patterns for the polished TiB$_2$ substrate before the test and for the Al/TiB$_2$ interface of the cross-sectioned couples after the sessile drop tests

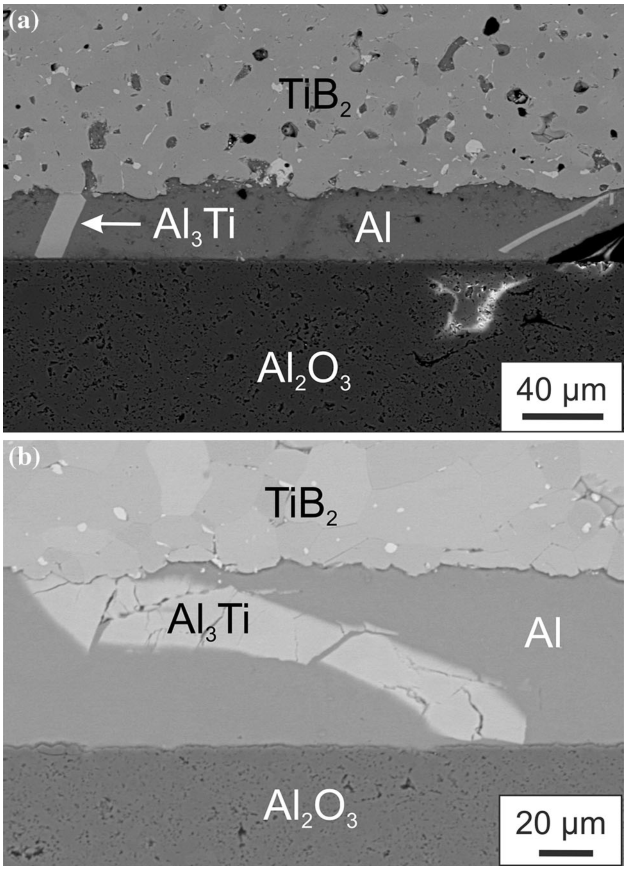

Fig. 10 Aluminium-filled gap between the TiB$_2$ substrate and the Al$_2$O$_3$ support after the sessile drop tests carried out for 15 min at **a** 1000 °C and **b** 1400 °C

diagram [34] and the high-temperature interaction between liquid Al and TiC ceramic [35]. It is interesting that Al$_3$Ti plate-like crystals were found in the Al-filled gap between the TiB$_2$ substrate and the Al$_2$O$_3$ support already in the experiments conducted starting from 1000 °C (Fig. 10). Probably, this is due to an enhanced oxidation of the TiB$_2$ surface contacting Al$_2$O$_3$ support upon heating and subsequent dissolution of TiO$_2$ in the liquid Al. The presence of Al$_3$Ti in the Al-filled gap at 1000 °C in our experiments correlates with the results reported in [31], where Al$_3$Ti plate-like crystals were formed in the Al–TiO$_2$ system after the tests carried out at 950 °C and higher temperatures.

Summary

The wetting behaviour and interfacial interactions between liquid Al and TiB$_2$ ceramic have been studied by the sessile drop method in the temperature range from 700 to 1400 °C. The interfacial microstructure of solidified Al/TiB$_2$ couples after high-temperature tests has been characterised by scanning electron microscopy, energy-dispersive X-ray spectroscopy and X-ray diffraction. For the classical sessile drop method, the onset of wetting of TiB_2 by liquid Al is observed at a rather high temperature (1030 °C) after deoxidizing the droplet surface. Capillary-purified aluminium in the dispensed drop technique starts wetting of TiB_2 at about 750 °C. The wetting is notably improved by continuous heating and changes by spreading over the TiB_2 ceramic at about 1000 °C. Complete wetting is also observed after isothermal annealing at 1000 °C for about 8 min and is significantly accelerated with increasing temperature.

No reaction between liquid Al and TiB_2 has been observed. However, beginning from 1000 °C, liquid Al penetrates the TiB_2 substrates by filling the intergrain pores as well as the grain boundaries at higher temperature. It extends up to about 250 µm after the sessile drop test carried out for 15 min at 1400 °C. Titanium aluminide Al_xTi, aluminium carbide Al_4C_3 and Al_2O_3 particles found at the Al/TiB_2 interface are supposed to originate from the reaction of liquid Al with TiO_2 and TiC impurities existing on the surface and in the bulk of the TiB_2 ceramic.

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