

Generation of isothermal spreading data and interfacial energy data for liquid reactive metals on ceramic substrates: the copper–titanium/alumina system

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The sessile drop technique is frequently used to evaluate the wettability and spreadability of liquid metals on ceramic substrates. In this study, the spreading kinetics of copper-20 wt % titanium alloys on polycrystalline alumina were evaluated based on measurements of spreading radius versus time. The process of spreading was monitored by an *in situ* video recording system. The tests were performed using three different initial metal configurations. It was found that conventional sessile drop testing configurations cannot be used to generate isothermal spreading kinetics data because of significant spreading during the heat-up cycle from the solidus temperature to the test temperature. An improved sessile drop technique was developed which eliminated the non-isothermal experience by introducing the liquid copper to the solid titanium/alumina at the desired testing temperature. Using this technique, only a few seconds of data were lost (while the liquid copper dissolved the solid titanium). Because very limited interfacial energy data exist for the copper–titanium/alumina system, especially at higher titanium concentrations, the equilibrium contact angle, the solid–liquid interfacial energy, and the work of adhesion from 1000 to 1300 °C are also presented.

1. Introduction

The wetting and spreading of liquid metals on ceramic substrates are important in ceramic–metal joining, composite processing and in the manufacture of electronic devices. In many of these applications, the metal alloy is chosen so that the wettability and adhesion of the liquid metal on the ceramic substrate are improved by the reaction of one of the metal components with the ceramic to form a new interfacial product [1]. The interfacial reaction mechanisms that govern the wetting and spreading of liquid reactive metals in contact with ceramic bodies are not clearly understood.

The sessile drop technique is frequently used to study the spreadability and wettability of liquid metals on ceramic and metal substrates [1–16]. In many of these studies, the contact angle is measured as a function of time. The wettability is then evaluated based on the minimum final contact angle or the time required to reach the final contact angle [1–6, 9–13, 16, 17]. Similarly, the spreadability is evaluated based on the change in the solid–liquid contact area (or contact radius) with time [4, 7, 8]. In these analyses, the relative spreadability is determined from the maximum contact area (or radius) and the time required to reach the maximum spreading area (or radius). If a liquid drop of known volume assumes a geometry which can be approximated as a spherical

cap, the contact angle can be calculated from the spreading radius and vice versa [15, 18, 19]. Thus, contact angle and contact radius (or area) measure similar phenomena for a spherical cap geometry.

In sessile drop experiments involving a non-reactive liquid on a ceramic substrate (i.e. copper or silver on alumina or silicon carbide [10, 20]), the equilibrium contact angle is attained in a few seconds. In this situation, the information obtained from this test consists of either the final contact angle or the final solid–liquid interfacial contact radius. Thus, the rate of sample heating from room temperature to the testing temperature and the total isothermal holding time are not important (except for holding times that are sufficiently long to cause metal volatilization and drop volume loss).

However, when a reactive metal is placed on a ceramic substrate, both the isothermal holding time and the sample heating rate are important. In general, when a reactive metal liquid is placed on a ceramic substrate and held at a constant temperature, the contact angle decreases and the spreading radius increases with time until constant values are reached. Consequently, these tests must be conducted for longer times compared to similar non-reactive metals if the equilibrium contact angle or spreading radius are desired. In reactive systems, spreading kinetics data

are needed to understand spreading mechanisms. Spreading radius, spreading area or contact angle versus time are commonly measured and interpreted [2, 4–12, 17]. However, if the rate of spreading is related to the rate of reaction product formation, significant non-isothermal spreading can occur during the heating cycle, and the generation of isothermal spreading kinetics data becomes almost impossible without very rapid heating rates.

Most sessile drop techniques for metal alloys on ceramics use one of two starting sample configurations. Either the metal is pre-alloyed to a given composition [7, 8, 17] or two or more pure starting materials are either stacked [2, 3, 12] or compacted as powders [13] so that, when the metal melts, the desired composition is achieved. For all of these techniques, the liquid metal is in contact with the ceramic substrate before the isothermal testing temperature is reached. Depending upon the heating rate and the difference between the solidus temperature and the testing temperature, an important fraction of the isothermal spreading data may be lost.

Ideally, the non-isothermal spreading would be eliminated by placing the liquid metal upon the ceramic substrate at the desired test temperature. Fujii *et al.* [5] were able to drop liquid aluminium on to boron nitride at the desired test temperature by using a back pressure to push an aluminium drop on to a boron nitride substrate. Their primary goal was to break the oxide film on the liquid aluminium surface before testing, but this experimental technique also resulted in complete (starting at $t = 0$ s) isothermal data of contact angle versus time. Similarly, Ip *et al.* [16] placed liquid aluminium upon calcia and alumina using a graphite plunger arrangement. Using this method, only the contact angle could be reported because control of the liquid metal volume was difficult.

If the oxide film on the liquid metal is not a factor in spreading, the metal alloy could be placed in a crucible, heated to the testing temperature and then poured on to the ceramic substrate. However, these techniques have a major experimental complication. Most reactive metals (e.g. titanium) either react with or dissolve the crucible materials. Dissolution results in a depletion of reactive metal from the liquid alloy, and a decrease in the drop volume. These changes make test reproducibility extremely difficult. For liquid aluminium alloys, the oxide film barrier hinders reaction between the ceramic substrate and the liquid metal, but also prevents reaction between the liquid aluminium and the container material. Aluminium alloys of known volume and composition could thus be introduced to a ceramic substrate at the desired testing temperature, while reaction between most other reactive metals and their containers complicates the control of volume and composition.

In this study, a different experimental approach eliminated the non-isothermal spreading of copper-20 wt % titanium alloys on polycrystalline alumina. In the solid state, the reaction of titanium with alumina is relatively slow. If a piece of titanium is placed on an alumina substrate, heated to 1300 °C and then cooled back to room temperature, the titanium slides off the aluminium plate when it is tilted. Also, there is

very little discoloration of the alumina surface where the titanium had rested implying that no significant reaction occurred between the alumina and the solid titanium. Similarly, liquid copper does not react with most ceramics. Therefore, if liquid copper is introduced on to an alumina substrate which already holds solid titanium, no significant spreading will occur until the copper dissolves the titanium. By submerging solid titanium, which is already in contact with alumina at the desired test temperature, isothermal spreading kinetics data can be generated if the time for titanium dissolution in the copper is small.

2. Experimental procedure

The sessile drop technique was used to generate spreading kinetics data as a function of initial sample configuration for copper-20 wt % titanium alloys on alumina. The metal samples were either a copper block (99.99% purity) and titanium block (99.99% purity), or a piece of pre-alloyed copper-20 wt % titanium ($> 99\%$ purity). The initial weight of the metal sample was 1.20 ± 0.02 g and the nominal composition was 20.0 ± 0.05 wt % titanium. The alumina was polycrystalline Coors AD996 electronic substrate (99.6% purity).

A tube furnace with a viewing window was used to obtain *in situ* diameter measurements of the sessile drop (Fig. 1). This system recirculated argon through an oxygen scavenger to reduce the oxygen partial pressure to ca. 50 p.p.b. The tests were performed at an argon overpressure of ca. 10 kPa. The spreading of the drops was videotaped, and the spreading diameter was measured from the videotape by freezing frames on a Leco image analyser. These measurements, accurate to within ± 0.1 mm, were corrected to account for the non-spherical contact area. The average radius was calculated by multiplying the measured radius by a proportionality constant. This constant was the ratio of the final radius measured perpendicular to the viewing direction divided by the final radius measured in the viewing direction. The assumption of an elliptical contact area was consistent with experimental observations. A preferred spreading direction, probably related to the processing of the square plates, was observed (the maximum spreading radius was always oriented perpendicular to one of the plate edges).

The tests were performed using three different initial metal configurations. The first configuration was a pre-alloyed block of copper-20 wt % titanium placed directly on an alumina plate (Fig. 2a). The second configuration consisted of stacked titanium and copper blocks stacked on alumina plates (Fig. 2b). In the third configuration, titanium was placed upon the alumina substrate and the copper was placed between two alumina plates above the titanium (Fig. 2c). At the desired test temperature, the liquid copper fell on to the titanium and formed a copper-20 wt % titanium alloy. In one special case, the copper and the titanium were placed side by side and the furnace was tilted at the test temperature to induce copper-titanium contact. This test technique resulted in the same test configuration as in Fig. 2c.

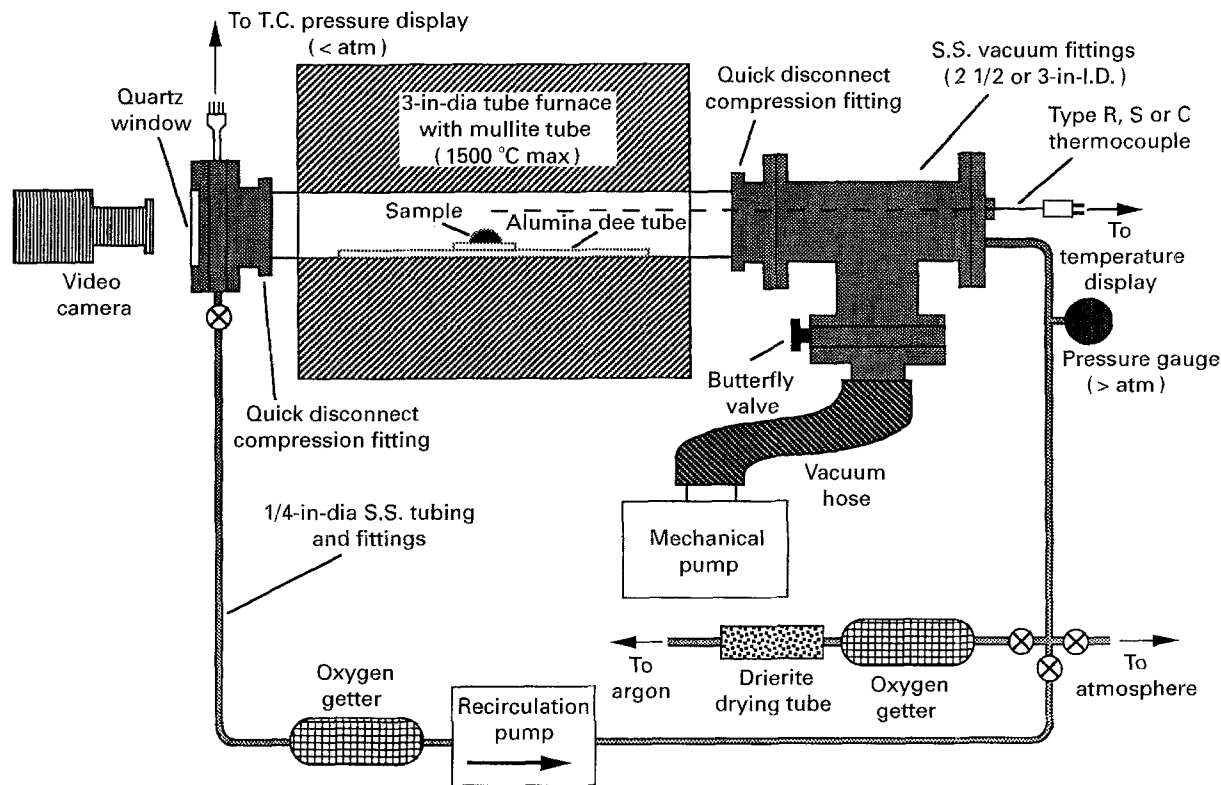


Figure 1 Schematic diagram of apparatus used to videotape the sessile drop spreading of copper-titanium alloys on alumina substrates.

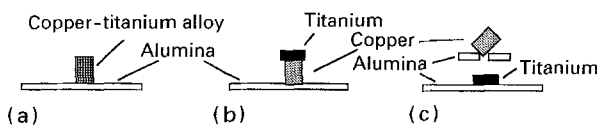


Figure 2 Initial room temperature starting configurations for sessile drop experiments (copper-20 wt % titanium alloys on alumina): (a) pre-alloyed copper-titanium on alumina; (b) stacked titanium on copper on alumina; and (c) titanium on alumina with copper in slot above titanium.

Tests were performed at 1000, 1100, 1200 and 1300 °C using the stacked configuration (Fig. 2b). Two tests were performed at 1200 °C for all three sample configurations to evaluate the effect of the starting configuration on the spreading kinetics. Each test lasted ca. 26 h (90 ks). Long testing times assured that the equilibrium spreading radii were obtained.

3. Results

The spreading radii for the stacked starting configuration (Fig. 2b), plotted as a function of time and temperature, are shown in Fig. 3. At each temperature, the spreading radius increased with time until a temperature-specific constant value was reached. The initial and final radii were smaller at lower temperatures, and the difference between the initial and final radii was larger at lower temperatures. These results are consistent with the data reported in the literature [2, 4-8, 11-13]. The spreading radius has been reported to increase with time to a constant value at an exponentially decaying spreading rate, while the contact angle exponentially decreases with time to a constant value. It has further been shown that an increase

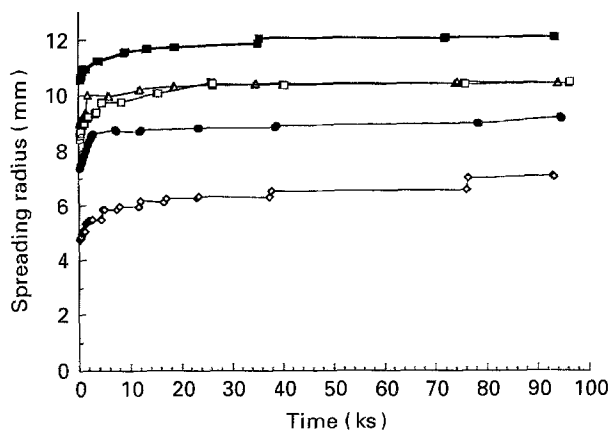


Figure 3 Spreading radius versus time for copper-20 wt % titanium alloys on polycrystalline alumina at 1000 (◇), 1100 (●), 1200 [□, (1); ■, (2)] and 1300 (△) °C. (The initial sample configuration was titanium stacked on copper on alumina.)

in temperature causes the initial and final spreading radii to increase (or correspondingly, the initial and final contact angles to decrease) while the differences between the initial and final radii (or contact angles) decrease.

Fig. 4 plots the spreading radius versus time for the same samples shown in Fig. 3, but includes the spreading which occurred during the heat-up cycle. The negative times on the plot represent the non-isothermal spreading observed from ca. 1100 °C to the testing temperature. All samples except the 1000 °C specimen showed significant spreading during the heat-up cycle from the point where the first liquid contacted the substrate to the isothermal holding temperature. Thus, significant isothermal spreading kinetics data were lost during the heat-up cycle.

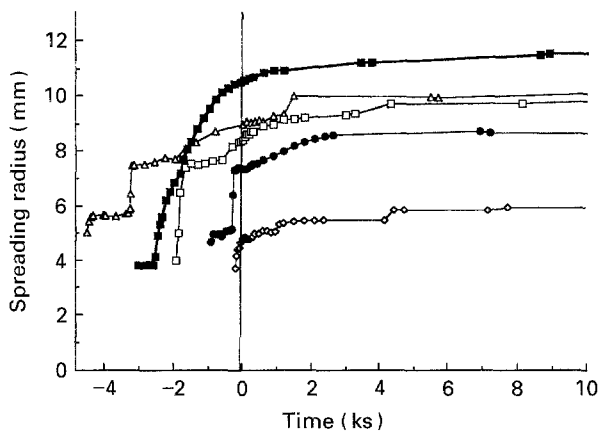


Figure 4 Spreading radius versus time for copper-20 wt % titanium alloys on polycrystalline alumina at 1000 (\diamond), 1100 (\bullet), 1200 (\square , (1); \blacksquare , (2)) and 1300 (\triangle) °C. The negative times represent the time required to heat the samples from 1100°C to the isothermal testing temperature. (The initial sample configuration was titanium stacked on copper on alumina. The negative times represent spreading which occurred during heating to the isothermal test temperature.)

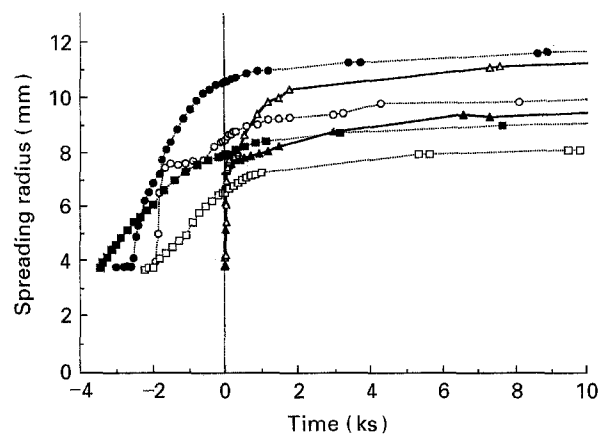


Figure 6 Spreading radius versus time for copper-20 wt % titanium alloys on polycrystalline alumina at 1200°C. The negative times represent the time required to heat the samples from the temperature at which the first liquid forms to the isothermal testing temperature. Three different starting configurations are plotted: (1) pre-alloyed Cu-Ti [\square , (1); \blacksquare , (2)] (see Fig. 2a); (2) Ti stacked on Cu [\circ , (1); \bullet , (2)] (see Fig. 2b); and (3) Cu (l) on Ti (s) [\triangle , (1); \blacktriangle , (2)] (see Fig. 2c). (The negative times represent (20) spreading which occurred during heating to 1200 °C.)

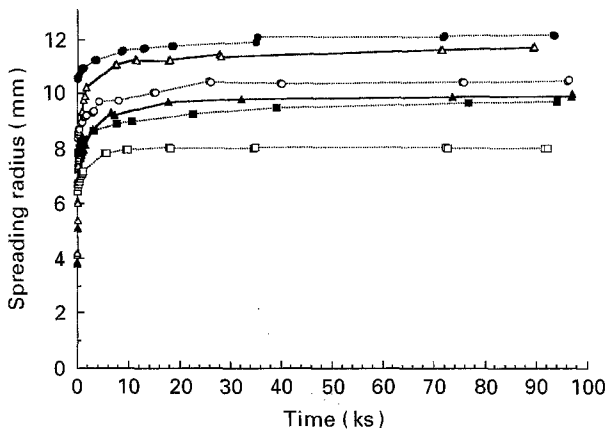


Figure 5 Spreading radius versus time for copper-20 wt % titanium alloys on polycrystalline alumina at 1200°C. Three different starting configurations are plotted: (1) pre-alloyed Cu-Ti [\square , (1); \blacksquare , (2)] (see Fig. 2a); (2) Ti stacked on Cu [\circ , (1); \bullet , (2)] (see Fig. 2b); and (3) Cu (l) on Ti (s) [\triangle , (1); \blacktriangle , (2)] (see Fig. 2c).

The spreading radii versus time plots for copper-20 wt % titanium on alumina at 1200 °C for the three different starting configurations are shown in Fig. 5. The spreading, which occurred before the sample reached 1200 °C is again shown as negative times in Fig. 6 (along with the short time spreading data at 1200 °C). The pre-alloyed sample formed a liquid and began spreading first, but the stacked sample actually spread more rapidly once a liquid formed. The configuration in which no liquid contacted the substrate until 1200 °C exhibited a much smaller spreading radius at $t = 0$ (upon reaching 1200 °C) than the other samples, but all of the samples spread to similar final radii. This observation supports the conclusion that significant isothermal spreading data are lost for the first two sample configurations (Fig. 2a and b).

4. Discussion

4.1. Spreading kinetics

The initial spreading kinetics can be qualitatively analysed based upon the time at which the first liquid

forms, and upon the titanium concentration of the liquid during the thermal cycle. Both factors are strongly influenced by the starting sample configuration.

4.1.1. Pre-alloyed copper-titanium alloy

According to the binary copper-titanium phase diagram (Fig. 7) [21], copper-20 wt % titanium should be completely molten at 885 °C. However, in this study melting was not observed at temperatures < ca. 1000 °C, probably because an oxide layer formed on the outside of the metal sample. When a liquid forms, it is not outwardly visible until the thermal expansion of the liquid drop breaks the oxide layer. Once this occurs, the liquid has the same composition as the initial solid (weight fraction, $X_{Ti} = 0.20$) which corresponds to a mole fraction of ca. 0.26. For this configuration, non-isothermal spreading was observed for between 2.2 and 3.4 ks while the sample was heated from ca. 1000 to 1200 °C.

4.1.2. Titanium stacked on copper

The melting of solid titanium in contact with solid copper was more complicated, but can still be qualitatively explained by reference to the binary phase diagram (Fig. 7). In this system, the first liquid formed should have had a composition between that of the intermetallics $Ti_2Cu + TiCu$. This liquid formed at the copper-titanium interface. As the copper and titanium dissolved into the liquid, the copper concentration increased until only liquid $TiCu$ remained and the liquid resolidified. In reality, the resolidification of the drop was not observed because of the slower dissolution kinetics of the solid copper by the liquid alloy relative to the dissolution of the solid titanium by the same liquid. The experimentally observed dissolution and melting path is shown schematically in Fig. 8.

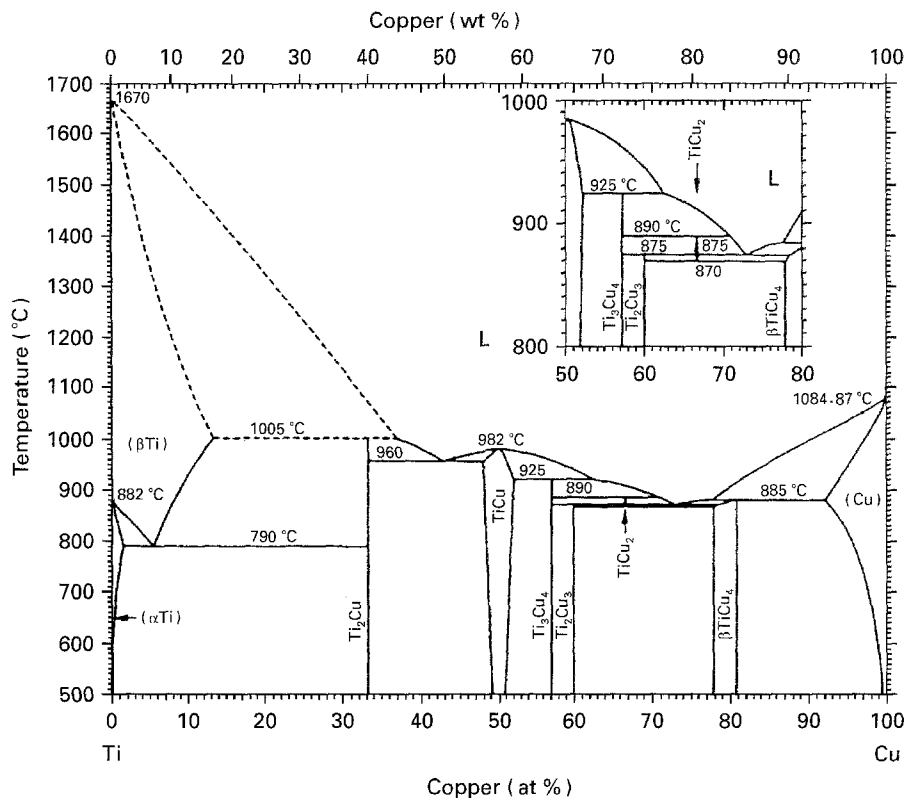


Figure 7 The copper–titanium binary phase diagram (Source: T. Massalski, “Binary alloy phase diagrams”, ASM International, Materials Park, OH, 1990), p. 1495.

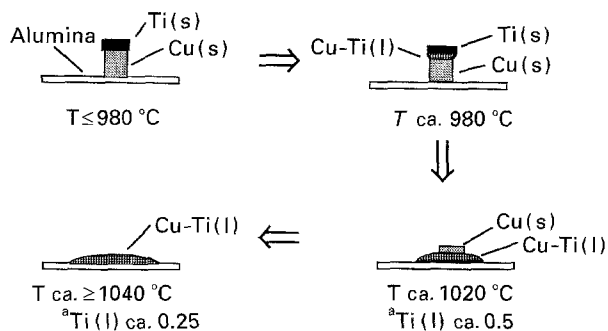


Figure 8 Schematic diagram of the changes that occur when a titanium/copper/alumina stack is heated from room temperature to 1200 °C.

First, a liquid formed between the copper and titanium at ca. 982 °C. The actual temperature at which this was observed was higher than that predicted from the phase diagram, because the liquid layer must be nearly 0.2 mm thick before it can be easily resolved by the camera. Next, the liquid front moved into both the copper and the titanium. The titanium dissolved more quickly than the copper, and eventually there was only a copper–titanium liquid capping a solid piece of copper. As more solid copper dissolved, the liquid volume increased and the liquid flowed off the solid copper. At this point, a titanium-rich liquid contacted the alumina substrate and began spreading (at ca. 1120 °C). Finally, at 1140 °C, the entire drop was liquid.

While the difference between the temperature at which liquid first contacted the alumina substrate and the isothermal holding temperature was less than that

for the pre-alloyed samples, the extent of spreading was somewhat greater. This result was unexpected because the time required to reach the isothermal holding temperature after the first liquid was formed was similar for both the stacked and pre-alloyed sample configurations. (The stacked samples equilibrated at 2.0 and 3.0 ks, while the pre-alloyed samples required 2.2 and 3.4 ks.) However, the stacked samples initially formed a titanium-rich liquid with a titanium mole fraction as high as 0.56. Assuming the activity to be approximately equal to the mole fraction, an initial titanium activity as high as ca. 0.56 was predicted for the stacked sample configuration. Furthermore, the activity decreased with time to ca. 0.26 as the drop became completely molten. In contrast, the pre-alloyed samples constantly maintained a titanium activity of 0.26 in the liquid. The higher initial titanium activity of the stacked sample configuration could explain the more rapid spreading observed during the heat-up cycle.

4.1.3. Liquid copper on solid titanium

When the liquid copper was introduced to the solid titanium at 1200 °C, the liquid was always at the desired temperature. (As an aside, the copper always melted at a temperature between 1085 and 1105 °C, crept down between the plates, and fell at 1200 °C. This observation supports the conclusion that it was the formation of an oxide layer rather than temperature measurement inaccuracies which led to the higher melting temperatures for the copper–titanium alloy.) The liquid was not at the desired composition until the solid titanium was completely dissolved, but

TABLE I Input values for interfacial thermodynamic calculations for copper-20 wt% titanium and polycrystalline alumina

T (°C)	V [22] (mm ³)	γ_{SV} [14] (mJ m ⁻²)	γ_{LV} [22] (mJ m ⁻²)	Starting configuration	r_{avg} (mm)
1000	175.0	1560	1445	Stacked	7.11
1100	176.4	1483	1422	Stacked	9.22
1200	178.7	1404	1398	Pre-alloyed	8.06
				Pre-alloyed	9.75
				Stacked	10.53
				Stacked	12.13
				Cu (l) on Ti (s)	11.73
				Cu (l) on Ti (s)	10.02
1300	179.6	1326	1374	Stacked	10.50

complete dissolution required only 10–15 s. In addition, the spreading was expected to be slower during dissolution, because the liquid was copper rich. This resulted in a lower titanium concentration and activity. Thus, the isothermal data lost was even less than the 10–15 s dissolution time, because the titanium-lean liquid should manifest slower spreading kinetics.

Overall, the best isothermal kinetics data were obtained by introducing the liquid copper to the solid titanium at the desired test temperature. This resulted in the loss of < 20 s of spreading data while the solid titanium dissolved into the liquid copper. Significant data (on the order of kiloseconds) was lost during the heat-up cycles for both the pre-alloyed and initially stacked metal sample configurations. The worst experimental condition occurred for the stacked metal samples where a high activity, titanium-rich liquid alloy was formed during heating.

4.2. Interfacial energies

Although conventional sessile drop tests cannot be used to analyse the isothermal spreading kinetics, these tests can be used to evaluate the equilibrium contact angle, the solid–liquid interfacial energy and the work of adhesion at long times, provided oxidation of the drop does not hinder spreading, and that depletion of the liquid metal by reaction and volatilization does not significantly decrease the drop volume. If a spherical cap geometry is assumed, the contact angle can be calculated based on the drop volume and the maximum radius of spreading [15, 18]. Because liquid metal alloy density data were not available, the volume of the copper–titanium drop was calculated from the mass using a “rule-of-mixtures” volume for the pure metals (based on the mass fractions of copper and titanium):

$$V_{Cu-Ti} \approx V_{Cu} + V_{Ti} = \left(\frac{M_{Cu}}{\rho_{Cu}} + \frac{M_{Ti}}{\rho_{Ti}} \right) m_{drop} \quad (1)$$

where V_{Ti} is the volume of liquid i , M_{Ti} is the mass fraction of i , ρ_{Ti} is the density of i , and m_{drop} is the total metal mass. The volume of a copper-20 wt % titanium sample weighing 1.2 g was calculated using the pure metal liquid densities given by Iida [22]. The volume of the starting samples and the maximum spreading

TABLE II Calculated interfacial thermodynamic properties for copper-20 wt% titanium on polycrystalline alumina

T (°C)	Starting configuration	θ (°)	γ_{SL} (mJ m ⁻²)	W_{ad} (mJ m ⁻²)
1000	Stacked	33.5	355	2650
1100	Stacked	16.2	117	2788
1200	Pre-alloyed	24.1	128	2674
	Pre-alloyed	12.9	42	2760
	Stacked	10.6	30	2772
	Stacked	6.6	15	2787
	Cu (l) on Ti (s)	8.1	20	2782
	Cu (l) on Ti (s)	12.9	41	2761
1300	Stacked	12.1	-17	2717

radii are given in Table I for the temperatures used in this study. For a spherical cap with a known volume, a given spreading radius has a fixed contact angle. For small contact angles, using the spreading radius to calculate the contact angle has been shown to be more accurate than a direct measurement of the contact angle [19].

If the liquid–vapour and solid–vapour energies are known, solid–liquid interfacial energy can be calculated at equilibrium using the Young equation:

$$\cos \theta = \frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}} \quad (2)$$

Similarly, if the liquid–vapour interfacial energy is known, then the work of adhesion, W_{ad} , can be calculated from the Dupre equation:

$$W_{ad} = \gamma_{LV}(1 + \cos \theta) \quad (3)$$

The solid–vapour interfacial energy (γ_{SV}) for alumina/argon from the work of Nikolopoulos [14] was used along with a “rule-of-mixtures” approximation for the liquid–vapour interfacial energy (γ_{LV}) calculated from the pure liquid data of Iida [22]:

$$\gamma_{LV}^T = X_{Ti}\gamma_{Ti}^T + X_{Cu}\gamma_{Cu}^T \quad (4)$$

where X is the mole fraction of (0.25 for Ti and 0.75 for Cu). The calculated values of γ_{SV} and γ_{LV} are given in Table I, and the resulting θ , γ_{SL} and W_{ad} values are given in Table II. The uncertainties in the volume calculation, the measured radius, and solid–vapour

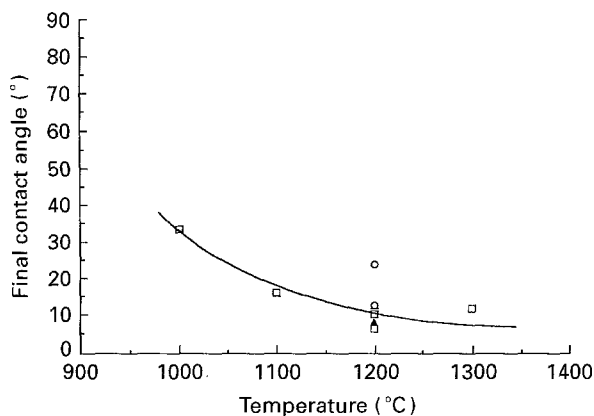


Figure 9 The equilibrium contact angle as a function of temperature for copper-20 wt % titanium on polycrystalline alumina. □, Stacked; ○, pre-alloyed; ▲, Cu (l) on Ti(s).

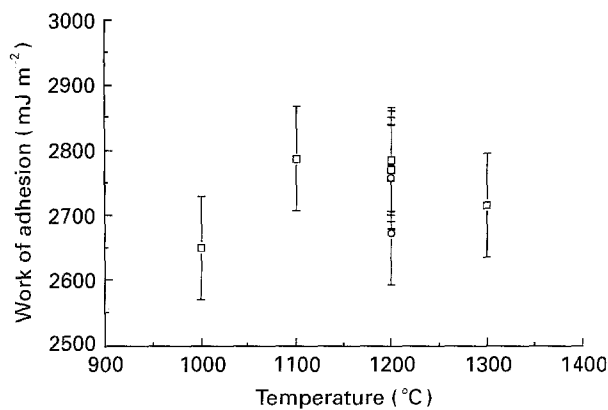


Figure 10 The work of adhesion as a function of temperature for copper-20 wt % titanium on polycrystalline alumina. □, Stacked; ○, pre-alloyed; ▲, Cu (l) on Ti(s).

and liquid–vapour interfacial energies lead to uncertainties of 60 to 80 mJ m^{-2} for the work of adhesion and the solid–liquid interfacial energy values, and 2–3° for the contact angles. The contact angle and work of adhesion values are plotted as a function of temperature in Figs 9 and 10, respectively. Unlike what was found in previous studies [1–5, 9, 10, 13], the contact angle does not decrease significantly with temperature and the work of adhesion does not increase significantly with temperature. Instead, these values are approximately constant from 1100 to 1300 °C. The contact angles of 7–34° are slightly lower than the values of 14–108° reported in the work of Naidich [1]. This result was expected, since the data reported by Naidich are for copper alloys containing 1–8 at % titanium (< 7 wt % titanium) at 1150 °C, while the samples for this study contained 20 wt % titanium (ca. 25 at %), and were studied at temperatures as high as 1300 °C. Work of adhesion values ranged from 2650 to 2790 mJ m^{-2} , while Naidich reported corresponding values from 1331 to 2522 mJ m^{-2} for 8 at % titanium at 1150 °C. The former values are in the range expected for high-titanium alloys.

With the exception of the 1000 and 1300 °C tests, the γ_{SL} values are very small (< 150 mJ m^{-2}) and positive. (The small negative value for 1300 °C reflects

the uncertainties in the calculation.) These results support the hypothesis that the formation of a TiO_x reaction product at the interface is the primary factor in the improved wetting and spreading of these alloys on alumina. Very low values of solid–liquid interfacial energy cannot be obtained between a metallic liquid and an ionocovalent solid because of the differences in bond character. However, oxides of titanium are known to take on a metallic character [23]. A liquid metal in contact with a metallic solid could have very similar bonding and a much lower interfacial energy.

The initial contact angles (upon dissolution at 1200 °C) were 105 and 97° for third sample configuration. These values suggest that only small improvements in the wettability of alumina are obtained by adding titanium to copper until a reaction occurs. Once reaction occurs, the contact angle is actually the contact angle of copper–titanium on a titanium oxide (with an alumina substrate outside of the radius of the drop), and not the contact angle of copper–titanium on alumina. The contact angle of high purity copper on polycrystalline alumina at 1200 °C and an oxygen partial pressure of ca. 10^{-5} Pa is 116° [18], which is only 11–19° higher than the value measured for copper–titanium on alumina at this temperature and oxygen partial pressure. The contact angle after the titanium–alumina reaction is complete is 90–100° lower than either of these angles.

5. Conclusions

This work showed that, conventional sessile drop configurations resulted in the loss of significant spreading kinetics data during the heat-up cycle for reactive metal liquids on ceramic substrates. The non-isothermal spreading of copper–titanium alloys on alumina was avoided by introducing the liquid copper to the solid titanium/alumina at the test temperature. Because relatively rapid spreading began shortly after the metal contacted the substrate, any sample configuration which allowed liquid metal–ceramic contact below the test temperature could not be used to generate true “isothermal” data. Without these data, the modelling of reactive metal spreading is complicated since both time and temperature are variables.

The determination of the equilibrium contact angle, the solid–liquid interfacial energy, and the work of adhesion were not significantly affected by the starting sample configuration. Regardless of the starting sample configuration, similar contact angles (7–24°), and work of adhesion values (2670–2790 mJ m^{-2}) were obtained for copper-20 wt % titanium on polycrystalline alumina at 1200 °C. The slightly lower energy values for the pre-alloyed copper–titanium samples may have been caused by sample oxidation during heating. However, this is not conclusive because of the relatively large experimental errors in these measurements.

By introducing the liquid copper to the solid titanium at the testing temperature, the contact angle for copper-20 wt % titanium on polycrystalline alumina was found to be 97–105° before the titanium reacted with the alumina.

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