

Surface tension of liquid Cu and anisotropy of its wetting of sapphire

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Abstract Precise surface tension data of liquid Cu are fundamental for studying its interaction with differently oriented single crystalline sapphire surfaces. For this reason, the surface tension of liquid Cu was measured covering a wide temperature interval of $1058\text{ °C} \leq T \leq 1413\text{ °C}$. To avoid contamination of the sample from contact with container walls, the measurement was performed contactlessly in an electromagnetic levitation furnace using the oscillating drop method. A fast digital CMOS-camera (400 fps) recorded top view images of the oscillating sample. From an analysis of the frequency spectrum the surface tension was determined. The measured surface tension of Cu was used to calculate interfacial energies from contact angles of liquid Cu droplets, deposited on the C(0001), A(11-20), R(1-102) and M(10-10) surfaces of sapphire substrates. These were measured by means of the sessile drop method at 1100 °C using a drop dispenser. Within the first minutes of contact with the sapphire substrates, the contact angles of liquid Cu droplets rise to their equilibrium values. From these, in addition to interfacial energies also works of adhesion were determined.

Introduction

In many technical applications like, for instance, in composite materials or microelectronic devices, the adhesion at metal–metaloxide interfaces is crucial. Especially the Cu/ $\alpha\text{-Al}_2\text{O}_3$ (sapphire) interface is of interest, since it can be

applied in low-loss connections [1] and, on the other hand, the Cu/ $\alpha\text{-Al}_2\text{O}_3$ system can be employed as a model system for metal ceramic interfaces [2] and heterogeneous nucleation processes. Cu has a relatively low melting point and the substrate is essentially chemically inert.

Several experiments [3–9] demonstrate that the wetting behaviour of a liquid melt may depend on the crystallographic orientation of the substrate. Hence, we study the wetting behaviour of liquid Cu on different $\alpha\text{-Al}_2\text{O}_3$ surfaces and examine its orientation dependence. For this purpose, precise knowledge of the surface tension of the liquid is fundamental. In the present work, these data are measured. The other subject is the determination of wetting angles and interfacial energies of the system Cu/ $\alpha\text{-Al}_2\text{O}_3$.

Interfacial energy and work of adhesion

In non reactive wetting, force equilibrium is established when a liquid droplet is placed on a flat solid surface. A contact angle, θ , is observed at the boundary between the three phases, liquid, solid and vacuum (or gas). If forces vertical to the substrate surface can be neglected, the Young equation is valid [10]

$$\sigma_{\text{S,L}}^{(hklm)} = \sigma_{\text{S,V}}^{(hklm)} - \gamma \cos \theta \quad (1)$$

here, $\sigma_{\text{S,L}}^{(hklm)}$ and $\sigma_{\text{S,V}}^{(hklm)}$ are the energies of the solid–liquid and the solid–vacuum interface, respectively. The superscript considers their orientation dependence by the Miller indices, h , k , l and m , of the crystallographic plane which forms the substrate surface. γ is the isotropic surface tension of the liquid.

The work of adhesion [10], $W_{\text{adh}} = \sigma_{\text{S,V}}^{(hklm)} + \gamma - \sigma_{\text{S,L}}^{(hklm)}$, is defined as energy which is required to reversibly separate the liquid from the solid creating two new surfaces from the

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original interface. W_{adh} is related to θ by the Young–Dupre equation

$$W_{\text{adh}} = \gamma(1 + \cos \theta) \quad (2)$$

The contact angle, θ , is measured by means of the sessile drop method using a drop dispenser. The advantage of using the dispensed sessile drop technique is that at the measurement temperature substrate and sample material can be equilibrated separately from each other. Hence, the measured contact angle is not distorted by the melting process itself or by inclosed contaminations at the interface [11].

Since we want to minimize the effect of gravity on the macroscopic contact angle, droplets of mass $M < 100$ mg are used for the sessile drop experiments. In doing so, almost ideally spherical cap shaped droplets are obtained. This fact makes it impossible to reliably determine the surface tension simultaneously from the solution of the Young–Laplace equation, since the uncertainty in determination of drop profiles rises enormously by decreasing the drop size [12]. To assure precise data, surface tension is measured in electromagnetic levitation, which is, in combination with the oscillating drop technique [13], particularly suited for the measurement of surface tension of liquid metals, which are highly sensitive to oxidation. Here the purity of the surface is of great importance [14, 15]. Using this technique, contamination of sample material due to contact with container walls is avoided.

Experimental

Surface tension measurement

Electromagnetic levitation

To prevent the sample material from contamination with oxygen, experiments are performed in a standard stainless steel high vacuum chamber, which is first evacuated and afterwards filled with 600 mbar He (purity: 99.9999 vol%) with 8 vol% H_2 acting as reducing agent. The almost spherical sample is positioned in the centre of a levitation coil to which an alternating current of typically 200 A is applied with a frequency of 300 kHz. Thus, an alternating inhomogeneous electromagnetic field is produced, which induces eddy currents inside the electrically conductive sample. These currents interact with the field such that a Lorentzian force establishes which compensates for gravitation of the sample. Due to ohmic losses the sample is also heated and molten. As levitation and heating are realized by the same coil, the sample must be cooled by a laminar flow of He/8%- H_2 gas to adjust a certain desired temperature.

The temperature of the sample is measured by a pyrometer focussed on the sample from the top. The pyrometer output signal has to be recalibrated with the known liquidus temperature, T_L , of the sample. The detailed calibration procedure is described in literature [16]. This calibration is done under the assumption that within the operating wavelength range of the pyrometer the emissivity of the sample material is independent of temperature. For most liquid metals this is a good approximation [17].

Oscillating drop method

The surface tension, γ , is determined by means of the oscillating drop method [13] from the frequencies of surface oscillations.

A surface which is deformed relative to its rest position performs oscillations around it. This is due to the surface tension acting as a restoring force. In terrestrial electromagnetic levitation, the equilibrium shape of the sample deviates from a sphere and is slightly elongated at the bottom. For this reason, the Rayleigh frequency [18], ω_R , of a spherical sample which is proportional to $\gamma^{1/2}$ splits up into a set of five frequencies, ω_m , belonging to the different oscillation modes with $m = -2, -1, 0, 1, 2$. From these frequencies, one can derive γ by means of the Cummings and Blackburn formula [19]

$$\gamma = \frac{3M}{160\pi} \sum_{m=-2}^2 \omega_m^2 - 1.9\Omega^2 - 0.3\left(\frac{g}{a}\right)^2 \Omega^{-2} \quad (3)$$

here M is the mass of the sample and a is its radius, calculated from M and from its density under the assumption of a spherical shape. The gravitational acceleration is denoted by g . The parameter Ω contains a correction for the magnetic pressure and can be calculated from the frequencies $\omega_{X,Y,Z}$, of the translational sample movements in X -, Y - and Z -direction: $\Omega^2 = 1/3(\omega_X^2 + \omega_Y^2 + \omega_Z^2)$.

The oscillation frequencies are determined from images of the oscillating drop. At each measured temperature, 4100 pictures are recorded with a fast C-MOS camera (400 frames per second) and afterwards evaluated by an edge detection algorithm. Considering the geometry of the different oscillation modes of the sample, the oscillation frequencies are obtained from the Fourier transform of the difference and the sum of two perpendicular radii of the sample [13].

Sample preparation

Samples with a diameter of about 6 mm were made of Cu metal with a purity of 99.999% (metals basis). Mass adjustment and surface cleaning were achieved by grinding

and ultrasonic treatment. Oxygen contaminations were removed by heating the samples to about 1500 °C until no bright oxide spots could be observed on the surface. After this alloying procedure and the subsequent experiment, the relative mass loss was less than 1.0%.

Contact angle measurement

Sessile drop experiments

Contact angles are measured in a sessile drop apparatus (Fig. 1) in a pure Ar atmosphere (purity: 99.9999 vol%) of about 300 mbar in order to reduce evaporation of sample material and heat removal from the system. The sample material is heated in a drop dispenser separate from the substrate, which is positioned about 5 mm below the dispenser. It consists of an Al₂O₃ ceramic tube with a small hole of 1 mm at the tip, through which the sample material is pressed when the desired measurement temperature is reached by careful application of Ar-gas pressure. If there is a potential oxide film on the alloy, it breaks as soon as the pressure is large enough and a fresh and clean droplet of about 2–3 mm diameter leaves the nozzle. Right under the substrate support, a type C (W-5%Re/W-26%Re) thermocouple measures the temperature within the tube furnace, which heats both the substrate and the metal in the dispenser resistively by Mo wires. Through a horizontal hole in the furnace, the sessile drop is illuminated from the back and, at the other side, a fast digital camera (1280 × 1024 pixels, 500 fps) records silhouette images. The edge curve of the droplet is determined by an image processing algorithm and then fitted with Legendre polynomials. From the slope of this fit at the left and the right contact point with the substrate, the contact angle is determined as average of the contact angles at both sides.

Sample material

For each contact angle measurement a purged Cu metal piece of about 100 mg was put into the dispenser.

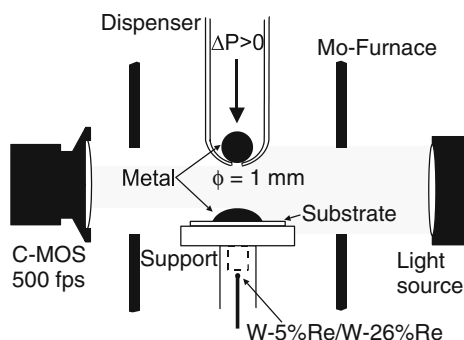


Fig. 1 Sketch of the sessile drop apparatus

Epipolished α -Al₂O₃ single crystals were used as substrates with differently oriented surfaces, namely the (0001) C-plane, (11-20) A-plane, (1-102) R-plane (all 5 mm × 5 mm × 0.5 mm) and (10-10) M-plane. The M substrates were broken away from a 2 in. wafer of 0.4 mm thickness. The average roughness and the maximum roughness of the substrates were <0.5 nm and <3 nm, respectively, other elements occurred with a concentration of <23 ppm.

These substrates were cleaned by rinsing with isopropanol before putting them into the sessile drop furnace, heating them to the measurement temperature of 1100 °C and annealing for at least 0.5 h.

Results

Surface tension measurement

Surface tension was measured for liquid Cu [20] in a wide temperature range of 1058 °C ≤ T ≤ 1413 °C. The obtained surface tensions, $\gamma(T)$, are plotted versus temperature in Fig. 2. Apparently, $\gamma(T)$ can be described as linear function of temperature with negative slope and the following equation can be fitted:

$$\gamma(T) = \gamma_L + \gamma_T(T - T_L) \quad (4)$$

here the parameters γ_L and γ_T denote the surface tension at liquidus temperature and the temperature coefficient, respectively. The fit yields $\gamma_L = (1.30 \pm 0.01) \text{ N m}^{-1}$ and $\gamma_T = (-2.64 \pm 0.9) 10^{-4} \text{ N (m K)}^{-1}$. The scatter of the measured surface tension data is only ±2.3%.

Contact angle measurement

After the droplet is dispensed, it touches the surface of the substrate and vibrates strongly conserving the kinetic energy of the falling drop. The contact angle oscillates

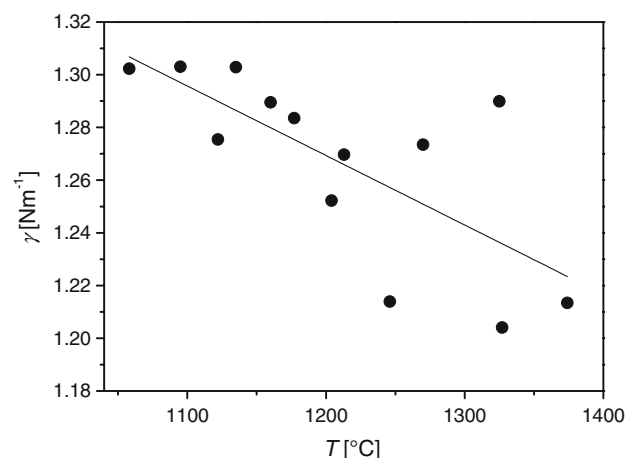


Fig. 2 Surface tension, γ , of liquid Cu versus temperature T

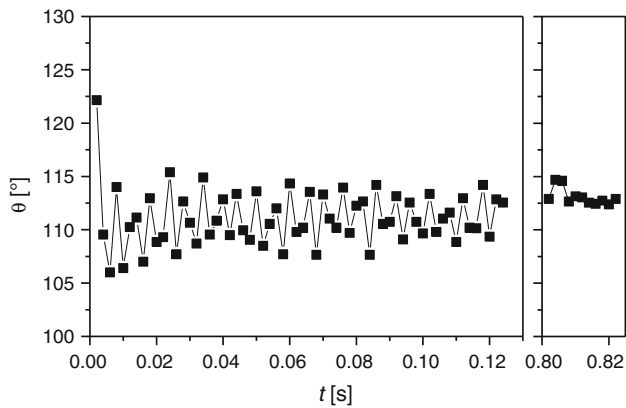


Fig. 3 Contact angle, θ , of liquid Cu on sapphire R-plane: shown is the short time evolution within the first second at 1100 °C

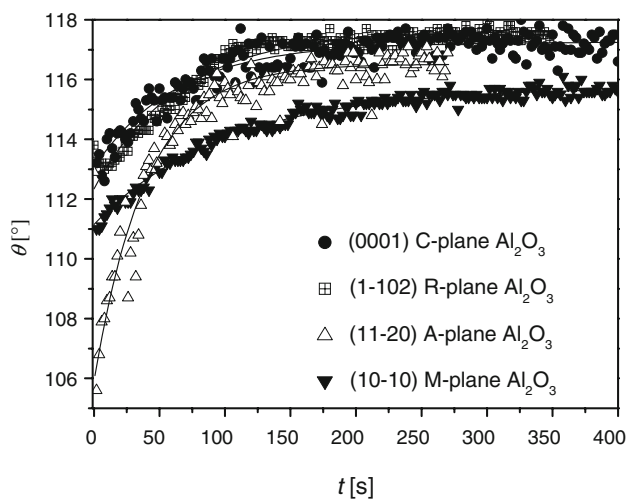


Fig. 4 Contact angle, θ , of liquid Cu on the different sapphire surfaces: shown is the time evolution within the first minutes after the dispensing process at 1100 °C

around its equilibrium value which is shown in Fig. 3 for the system Cu/R-plane (1-102)–sapphire at 1100 °C. Within approximately 1 s the oscillations decay by viscous damping and the contact angle remains stable at about 113° for this short timescale.

A longer observation period is considered in Fig. 4, where the time evolution of the measured contact angles is shown for the different sapphire surfaces C(0001), R(1-102), A(11-20) and M(10-10) at 1100 °C. Starting from a lower value, the contact angle increases for all substrate surfaces until it is constant after approximately 200 s. The parameters of this limited exponential growth of θ , $\theta(t) = \theta_\infty - (\theta_\infty - \theta_0)e^{-t/\tau}$, are given in Table 1. The initial value of the contact angle, θ_0 , lies between 106° and 113° ± 5°. For all surfaces, the upper limit contact angle, θ_∞ , is, within the uncertainties, approximately the same: 117° ± 5°.

Table 1 Fitting parameters of the limited exponential growth of $\theta(t)$ for the different liquid Cu/ α -Al₂O₃ interfaces at $T = 1100$ °C. θ_0 is the contact angle at $t = 0$, θ_∞ is its upper limit and τ is the time constant of the growth

	C(0001)	R(1-102)	A(11-20)	M(10-10)
θ_0 [°]	113 ± 5	112 ± 5	106 ± 5	111 ± 5
θ_∞ [°]	117 ± 5	118 ± 5	117 ± 5	116 ± 5
τ [s]	63.7 ± 0.8	65.9 ± 0.6	40.0 ± 0.4	100.7 ± 1.2

Discussion

Surface tension

The surface tension data show that in electromagnetic levitation the measurement conditions are good enough to avoid a significant dissolution of oxygen in liquid copper. Otherwise the increasing adsorption of oxygen in liquid copper would lead to a flattening or even reversal of the slope of the surface tension when measurement temperatures are lowered. The measured temperature dependence of the surface tension is in agreement with literature values [21].

Contact angle

The purpose of our dispensed sessile drop measurements was to find the true contact angle of pure liquid Cu on the differently oriented sapphire substrates, before any possible contaminations of the contact angle set in, like for instance from evaporation of sample material, oxidation of the interface or ridging at the triple line.

The effect of oxygen on the Cu/ α -Al₂O₃ system is observed in literature, leading to a decrease of the contact angle for increasing p_{O_2} (Table 2), which is either stepped at an oxygen partial pressure of about 10⁻¹¹ bar (from 130° ± 2° to 119° ± 3° at 1092 °C) [22], or linear in a range of 10⁻¹⁸ bar to >10⁻³ bar (from 124° ± 1° to about 90° at 1200 °C [23], or from about 127° to 100° at 1100 °C [11]), or it shows a steep decrease for $p_{O_2} > 10^{-6}$ bar (from contact angles around 115° to values below 90°) [24]. In case of high p_{O_2} , the formation of an interfacial layer of Cu₂O or even CuAlO₂ is suggested [23] and observed [24]. Our samples were processed in an Ar ambient with $p_{O_2} < 10^{-6}$ bar and no formation of a microscopic interfacial layer could be observed. On the other hand, even for low p_{O_2} , a formation of Cu–oxygen clusters in the liquid is suggested, leading to an adsorption of oxygen at the interface [25, 26].

Theoretically, the contact angle can also be changed by pinning at nm size ridges that could form within seconds at 0.2–0.5 T_m (melting temperature of sapphire: $T_m = 2053$ °C). The formation of such ridges (up to 40 nm

Table 2 Literature values of the decrease of θ with respect to ambient p_{O_2}

Kind of decrease	Low p_{O_2} [bar]	θ at low p_{O_2} [°]	High p_{O_2} [bar]	θ at high p_{O_2} [°]	T [°C]	Reference
Stepped	10^{-11}	130 ± 2	10^{-11}	119 ± 3	1092	[22]
Linear	10^{-15}	124 ± 1	10^{-3}	≈ 90	1200	[23]
Linear	10^{-18}	≈ 127	10^{-4}	≈ 100	1100	[11]
Linear						
Flat	10^{-15}	>130	10^{-6}	≈ 115	1100	[24]
Steep	10^{-6}	≈ 115	10^{-3}	≈ 90	1100	[24]

height) is observed for Cu on sapphire at 1150 °C after processing for 1–1.5 h, leading to a macroscopic contact angle of 114° for both advancing and receding droplets [27]. From this behaviour, Saiz et al. deduct that the ridge is small compared to droplet radius and the macroscopic contact angle corresponds to Young's contact angle θ .

Evaporation of Cu from a pinned droplet would cause a decrease of θ due to the volume loss until the triple line breaks away and θ jumps to a higher value, as it is seen for Al/sapphire [27].

Hence, the increase of θ , which we observe, cannot be explained by these processes.

From Young's equation 1 one can see, that for $\theta > 90^\circ$ an increase of the contact angle occurs, if $\sigma_{S,L}$ increases or γ or $\sigma_{S,V}$ decreases. Another possibility is that the increase of $\sigma_{S,L}$ compensates for an increasing γ or vice versa.

Since pure metals hardly wet oxide surfaces, a decrease of $\sigma_{S,V}$ due to adsorption of Cu on the substrate surface can be neglected.

A more favourable explanation of the behaviour of θ is given by the increase of $\sigma_{S,L}$: As discussed, in literature, the observed effect of oxygen on the Cu/Al₂O₃ system is a decrease of the wetting angle. This is explained by adsorption of oxygen at the liquid surface and the solid–liquid interface, minimizing the free energy of the system. This effect leads to a decrease of both $\sigma_{S,L}$ and γ , in which the decrease of $\sigma_{S,L}$ overcompensates the one of γ , due to additional coulombic interactions of the Cu–oxygen clusters with the substrate [25].

If, in our experiment, the dispensed drops were oversaturated in oxygen content with respect to ambient p_{O_2} ,

dissolved oxygen would desorb to the vapour phase, leading to an increase of θ . From the diffusion constant of oxygen in liquid Cu at $T = 1100$ °C, D_{O}^{Cu} (1100 °C) = $5.5 \cdot 10^{-9}$ m²/s [28] one can estimate an oxygen diffusion time, t , from $t = r^2/2D_{O}^{Cu}$. Here r is the mean distance traveled within t and corresponds to the radius of the droplet, which is about 1 mm. We obtain a diffusion time, which is in the order of 100 s and hence coincides with the time constant τ of the increase of θ (Table 1).

The equilibrated contact angles of about $117^\circ \pm 5^\circ$ for the different substrate surfaces are in good agreement with the p_{O_2} dependent literature data [11, 22–24], suggesting 10^{-6} bar $< p_{O_2} < 10^{-12}$ bar.

Interfacial energy and work of adhesion

To evaluate Young's equation 1, the surface tension of liquid copper and the surface energies, $\sigma_{S,V}$, of the pure unoccupied substrate surfaces need to be known. The surface tension of liquid copper was measured in this work, but values for $\sigma_{S,V}$ need to be taken from literature. In [29], a comprehensive and thorough compilation of surface energies of different α -Al₂O₃ surfaces is given obtained by various theoretical and experimental techniques. There, the $\sigma_{S,V}^{(hklm)}$ are normalized with respect to the value of the C-plane. Although only few data [29, 30] on the temperature dependence of these relative surface energies are available, they appear to show no significant change with temperature. Hence, in a first attempt it is reasonable to assume that the $\sigma_{S,V}^{(hklm)}$ are constant.

Table 3 Parameters of the liquid Cu/ α -Al₂O₃ interfaces at $T = 1100$ °C. $\sigma_{S,L}^{(hklm)}$ were determined from literature values of $\sigma_{S,V}^{(0001)}$ [10, 31] and from $\sigma_{S,V}^{(hklm)*}$ [29], which denote relative surface

energies of the substrates, normalized with the surface energy of the C-substrate. Surface tension of Cu at 1100 °C: $\gamma_{Cu}(1100 \text{ °C}) = 1.31 \text{ Nm}^{-1}$

	C(0001)	R(1-102)	A(11-20)	M(10-10)
θ_∞ [°]	117 ± 5	118 ± 5	117 ± 5	116 ± 5
W_{adh} [Jm ⁻²]	0.71 ± 0.10	0.69 ± 0.10	0.71 ± 0.10	0.73 ± 0.10
$\sigma_{S,V}^{(hklm)*}$ (at $T = 1600$ °C)	1	0.855 ± 0.017	0.974 ± 0.026	>1.008
$\sigma_{S,V}^{(hklm)}$ [Jm ⁻²]	1.13 ± 0.05	0.96 ± 0.07	1.10 ± 0.08	>1.14
$\sigma_{S,L}^{(hklm)}$ [Jm ⁻²]	1.72 ± 0.11	1.58 ± 0.12	1.69 ± 0.13	>1.71

For the C surface the absolute value of $\sigma_{S,V}^{(0001)}$ is obtained from experimentally determined literature data, recommended in review articles [10, 31]. These data can be linearly approximated by the following equation:

$$\sigma_{S,V}^{(0001)} = 2.14 \text{ Jm}^{-2} - 7.4 \cdot 10^{-4} \text{ Jm}^{-2} \text{ K}^{-1} \cdot T \quad (5)$$

From interpolation we determine $\sigma_{S,V}^{(0001)}$ at 1100 °C, at which the measurements were performed.

In Table 3, the applied data and the obtained interfacial energies are shown for different planes of sapphire. Additionally, the work of adhesion, W_{adh} , was determined. The uncertainties of these values can be estimated to be less than $\pm 0.1 \text{ Jm}^{-2}$.

The work of adhesion, W_{adh} , is virtually isotropic with respect to the three faces, C, R, A and M, since the contact angle is the same for all of them. Its value of about 0.7 Jm^{-2} is slightly higher than literature data [11, 22, 23] in the medium p_{O_2} range, since γ_{Cu} in this work is lower than the values used there. The normalized values of the surface energies, $\sigma_{S,V}^{(hklm)*}$, of the C-, A- and M-substrates vary only slightly, but for the R-plane it seems more than 15% lower than $\sigma_{S,V}^{M(10-10)*}$, and the same applies for the absolute values, $\sigma_{S,V}^{(hklm)}$, calculated from the normalized ones.

Within their uncertainties, the interfacial energies $\sigma_{S,L}^{(hklm)}$ of liquid Cu with the C-, A- and M-substrates show no pronounced anisotropy, $\sigma_{S,L}^{(hklm)} = (1.7 \pm 0.1) \text{ Jm}^{-2}$. The interfacial energy of Cu with the R-substrate, $\sigma_{S,L}^{R(1-102)} = (1.58 \pm 0.12) \text{ Jm}^{-2}$, is smaller by 7%.

Conclusions

Precise surface tension data were measured for liquid Cu covering a wide temperature range. It was found, that γ decreases linearly with increasing temperature.

In performing dispensed sessile drop experiments, an increase of the contact angle within the first minutes was observed. After equilibration the same contact angle, $\theta = 117^\circ \pm 5^\circ$, was determined for liquid copper on C-, R-, A- and M-surfaces of sapphire. As the contact angle is isotropic, the work of adhesion is so as well. The solid–liquid interfacial energy of the system shows a slight anisotropy for R-plane sapphire.

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