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Wetting angle and surface tension of germanium melts on different substrate materials

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Abstract

The sessile drop technique has been used to measure the wetting angle and the surface tension of molten germanium (Ge) on various substrate materials. Sapphire, fused silica, glassy carbon, graphite, SiC, carbon-based aerogel, pyrolytic boron nitride (pBN), AlN, Si₃N₄, and CVD diamond were used as substrate materials. In addition, the effects of different cleaning procedures and surface treatments on the wetting behavior were investigated. The highest wetting angles with values around 170° were found for pBN substrates under active vacuum or with a slight overpressure of 5 N argon or forming gas (2% hydrogen in 5 N argon). The measurement of the surface tension and its temperature dependence for Ge under a forming gas atmosphere resulted in $\gamma(T) = 591 - 0.08(T - T_m)[10^{-3}\text{N/m}]$. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

Precise knowledge of material parameters is more and more important for improving crystal growth processes. Two important parameters are the surface tension and the wetting angle. Both parameters determine the meniscus shape in a

variety of methods, e.g. Czochralski, EFG, floating-zone, and detached Bridgman (dtB) growth. Two methods exist to measure wetting angles and surface tension simultaneously, the capillary rise [1] and the sessile drop [1,2] methods. The sessile drop method is more accurate, especially for high temperatures, and was used for the investigations presented in this paper. Surface tension measurements of Ge have been reported in the literature and other methods (maximum bubble pressure [1], ring weight [1], Wilhelmy slide [1], ring depression [3], oscillating jet [1], and levitated (oscillating)

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drop method [4,5]) can be used for its determination. In general, the levitated drop method gives the best results for the surface tension alone due to the fact that it is a crucible free method, and it has recently been used to determine material parameters for liquid Ge [5], including the surface tension. However, no systematic investigation of the wetting angle of germanium with different crucible materials is known to the authors.

The specific background for the experiments was the selection of a suitable ampoule material for the detached growth process of Ge–Si alloy crystals. There are two main factors influencing detached growth:

1. According to Zemskov et al. [6] and Duffar et al. [7], detached growth can be achieved if the sum of the wetting angle θ between melt and crucible and the growth angle α between the crystal and melt is equal to or exceeds 180° .
2. Detachment can also be achieved when $\theta + \alpha < 180^\circ$ if there is a higher pressure in the volume below the meniscus [8]. This pressure difference can either be achieved by artificial means, or according to a theory by Wilcox [9,10], by the segregation of gas in the melt at the growing interface.

In both cases, the knowledge of the wetting angle with the crucible material during growth is essential to achieve or control the detached growth of semiconductor crystals. A starting point for the investigations on detached growth of Ge–Si crystals was the properties of Ge, since Ge is known to show detachment [11] during the Bridgman growth. This paper therefore focuses on the wetting angle and the surface tension of molten Ge on different substrate materials, measured between the melting temperature and 1090°C . A manuscript, which gives the results of subsequent investigations on Ge–Si melts, is in preparation [12].

2. Experimental procedure

Using the sessile drop technique, wetting angles and surface tension can be determined

simultaneously.¹ The shape of the drop is given by the Laplace capillary equation:

$$\gamma \left(\frac{1}{R_1} + \frac{1}{R_2} \right) = \Delta P, \quad (1)$$

where γ is the surface tension of the material, R_1 and R_2 are the principal radii of curvature at a point of the liquid surface and ΔP is the capillary pressure. The wetting angle θ is determined by calculating the tangent of the curve at the sample–substrate interface. In general, θ is determined by the three interfacial energies σ , the solid–gas energy σ_{sg} , the solid–liquid energy σ_{sl} and the liquid–gas energy σ_{lg} , as expressed in the Young equation [13]:

$$\cos(\theta) = \frac{\sigma_{\text{sg}} - \sigma_{\text{sl}}}{\sigma_{\text{lg}}}. \quad (2)$$

The Young equation assumes that the substrate surface is completely smooth, homogeneous, isotropic, and non-deformable. All of these conditions are violated in real world systems, leading to wetting angle hysteresis. Wetting angle hysteresis is the difference of the angles that can be measured for a liquid that is either receding or advancing on the substrate. It is associated with local minima of the free energy of the drop as a function of the wetting angle [13]. However, no significant difference in the wetting angle could be found for samples that started with a thin, large diameter cylinder (thus resulting in a receding wetting angle upon melting) and samples that started from a thick, smaller diameter cylinder (thus resulting in an advancing wetting angle upon melting). The surface tension can be determined by fitting a curve based on the Laplace capillary equation (1) around the drop and computing the curve parameters.

2.1. Experimental setup

Fig. 1 shows a schematic drawing of the sessile drop apparatus as it was used in the experiments. A heat pipe (No. 2 in Fig. 1) was used inside the furnace to achieve isothermal conditions over the whole length of the sample.

¹The influence of line tension [8], which is only important for very small drop sizes, is neglected here.

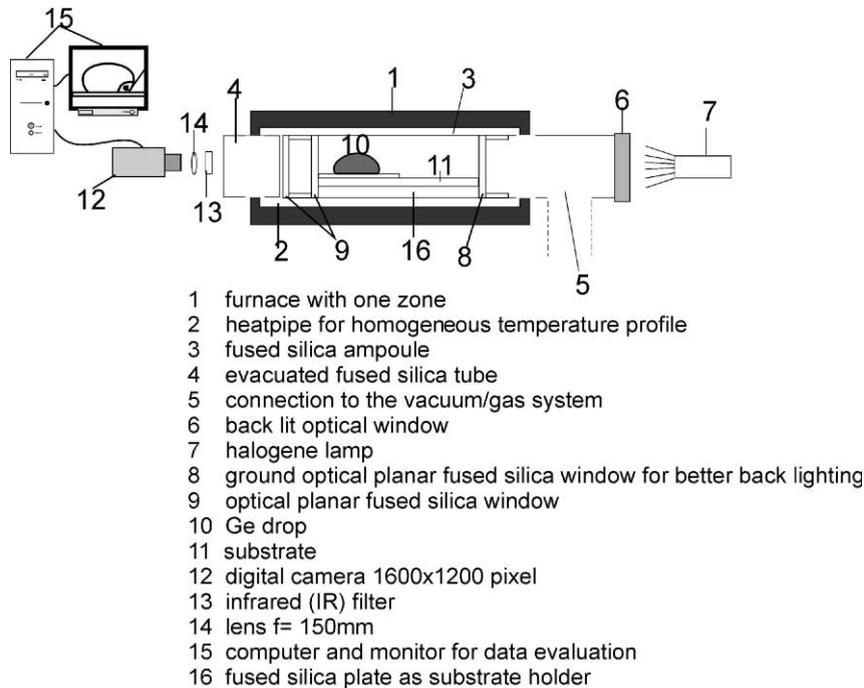


Fig. 1. Schematic drawing of the sessile drop setup reaching temperatures up to 1100°C.

Sapphire, fused silica, glassy carbon, graphite, SiC, carbon-based aerogel, pyrolytic boron nitride (pBN), AlN, Si₃N₄, and CVD diamond were used as substrates for these measurements. For fused silica and sapphire, different surface treatments were also used (see Table 1). Sample and substrate materials were located inside a fused silica ampoule (600 mm long, 28 mm ID, No. 3 in Fig. 1) sealed on one side with an optical planar window (see Fig. 1 No. 9). It was open on the other side to connect to the vacuum/gas system (No. 5 in Fig. 1). Experiments were performed either under dynamic vacuum (9×10^{-6} mbar, measured about 200 mm from the ampoule), under a slight overpressure (1030–1040 mbar) of 5 N argon, or under a slight overpressure of forming gas (2% H₂, 98% Ar). The gas line was fitted with an Oxisorb[®] cartridge to further reduce the residual oxygen content of the gas. The substrate holder was a fused silica plate (No. 16 in Fig. 1) that could be removed from the outer fused silica ampoule No. 3 in Fig. 1. The optical system consisted of an IR cutoff filter, a 150 mm f/9 process lens and a digital microscope camera with

1200 × 1600 pixels resolution and a chip diagonal of 12.15 mm (Nos. 13, 14 and 12 in Fig. 1, respectively). Typical magnifications were of the order of $m=0.7$, and the (diffraction limited) system resolution was 25 μm. The optical axis of the system was positioned slightly above the substrate surface, so that the drop and the substrate were viewed under a slight angle. In this way, one can view the shadow image of the drop and its reflection at the same time, resulting in an easy way to determine the contact line between drop and substrate by the angles between the two. An evacuated fused silica tube (No. 4 in Fig. 1) with optical planar windows at both ends is placed in front of the sample tube; one end is located outside the furnace and thus prevents optical distortions due to the hot air rising from the furnace front.

2.2. Sample preparation and experimental procedures

Before starting the experiments, all fused silica parts were cleaned in acetone/methanol and rinsed

Table 1
Surface treatment and cleaning procedures of substrates^a

Substrate	Cleaning procedure
Fused silica (Chemglas)	
Fused silica, HF fume etched	Dri-Contrad ^{®a} , acetone/methanol
Fused silica, sandblasted	
Fused silica, fire polished	Aqua regia
Sapphire (Saphikon)	
Sapphire, HF etched	Dri-Contrad ^{®a} , acetone/methanol
Sapphire, sandblasted	Acetone/methanol
CVD diamond (IAF Freiburg)	Dri-Contrad ^{®a} Acetone/methanol Baked out (1080°C) under dynamic vacuum
Glassy carbon (Graphite Die Mold)	Acetone/methanol Baked out (1080°C) under dynamic vacuum
Graphite (Graphite Die Mold)	Acetone/methanol Baked out (1080°C) under dynamic vacuum
SiC (SuperSiC [®] , Poco Graphite)	2:2:1 HF(50%):HNO ₃ (69%):H ₂ O
Aerogel (C-based) (DLR Cologne)	—
pBN (Performance materials)	Baked out (1080°C) under dynamic vacuum
AlN (Accuratus)	HCl (49%) Baked out (1080°C) under dynamic vacuum
Si ₃ N ₄ (Ceralloy 147)	Baked out (1080°C) under dynamic vacuum

^a Dri-Contrad[®] is a commercial cleaner for lab glassware.

with distilled water (13 MΩ resistivity). The treatment of the substrates differed according to the materials, see Table 1.

The germanium samples were cylindrical sections (8 and 12 mm diameter) cut from single crystalline rods (source: Eagle-Picher company optical grade, 4–40 Ω cm). The mass of the samples was typically 2–3 g. They were etched in an 18:8:5 (HNO₃(69%):CH₃COOH(100%):HF(49%)) etch for several minutes to remove impurities and oxide layers, and subsequently stored in deionized water. Right before the Ge sample was placed in the ampoule, it was etched again in a KOH solution (20–30 g KOH in 100 ml water) for 10 min

to remove residual oxides, and rinsed in 13 MΩ water. After loading the ampoule, it was inserted into the furnace and evacuated for several hours. The whole system was flushed several times with argon and/or forming gas before being heated up slowly to the melting temperature of Ge under dynamic vacuum. All samples were melted under dynamic vacuum to avoid the formation of bubbles below the drop. Once the melting temperature of the material was reached and the drop started to form, it was monitored by taking pictures every 5–15 min at 950°C to check the sample position and observe the change of the wetting angle and surface tension over time.

After approximately 60–90 min, the entire sample was molten and thermal equilibrium was reached. To determine temperature dependencies, the temperature was raised in 10 or 20 K intervals up to 1080°C or 1090°C. To ensure thermal equilibrium, pictures, usually in sets of four, were taken after 30–40 min waiting time. After reaching the maximum temperature, the temperature was reduced again until the melting point was reached. In some experiments, the sample was processed for longer times, up to a week, to investigate any changes in the wetting angle and/or surface tension due to slow reactions with the substrate.

2.3. Evaluation procedure

To evaluate the drop shape, the outline of the drop was traced with edge detection software. The resulting curve parameters (pixel x - and y -coordinates) were the input for an evaluation algorithm programmed by Tegetmeier [1] in Mathematica[®]. Additional inputs were the mass of the drop and the magnification (scale factor). An approximate value for the scale factor could be derived from the known diameter of the solid cylindrical starting material by taking an image of the solid sample.

The germanium drop, however, could move upon melting, changing the magnification slightly. Since the density of the drop is also calculated during the evaluation of the drop shape (assuming rotational symmetry), the literature value of the temperature dependent density of germanium from Ref. [5] was used for adjusting the scale factor. The program then fits the parameters of the theoretical curve to the image trace data and calculates the surface tension γ using Eq. 3,

$$\gamma = \frac{\rho g}{q} R_0^2 \quad (3)$$

with g = gravitational acceleration, ρ = density, R_0 = radius at the top of the drop, q = curvature of the drop at the sample–substrate transition.

The wetting angle is determined by calculating the tangent at the sample–substrate transition.

3. Results and discussion

3.1. Wetting angle

Table 2 shows a summary of the wetting angles of Ge on the different substrate materials. The * denotes a change of the wetting angle over time as

Table 2
Measured wetting angles on different substrate materials^a

	Substrate	Surface treatment	Atmosphere	Wetting angle (°)
O-based substrates	Fused silica	—	Vacuum	150–117*
		Sandblasted	Vacuum	155–128*
	Sapphire	Fume etched	Vacuum	156–131*
		Fire polished	Vacuum	148–131*
C-based substrates	Sapphire	Sandblasted	Vacuum/argon	150–119*
			Vacuum/argon	144–134*
	Graphite		Vacuum	166 ± 1
	Glassy carbon		Vacuum	157 ± 1
	CVD diamond		Vacuum	146 ± 1
N-based substrates	SiC		Vacuum	167 ± 2.5
	C-based aerogel		Vacuum/argon	173 ± 2.5
	Si ₃ N ₄		Vacuum/argon	157–136*
	AlN		Vacuum/argon	170–153*
	pBN		Forming gas	173 ± 3

^a Note: * denotes a change of the wetting angle over time due to reactions with the substrate material. The error for the wetting angles (given for the cases without a systematic change of the angle) is the standard deviation.

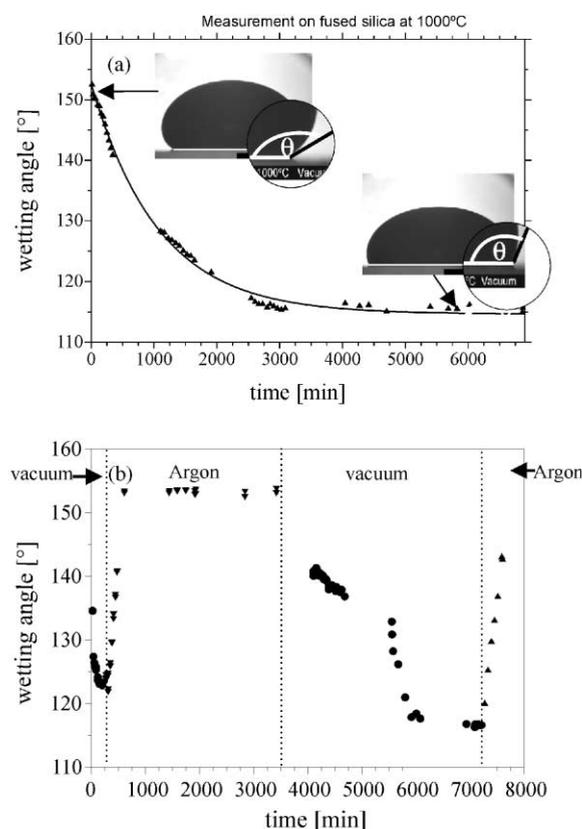


Fig. 2. Wetting angle of Ge on fused silica (a) under dynamic vacuum showing a decrease over time due to a reaction with the substrate and (b) showing the effect of a change between dynamic vacuum (●) and 1040 mbar argon (▼▲) on the wetting angle.

shown in Fig. 2a, indicating reactions with the substrate material. This happened for fused silica, sapphire, silicon nitride, and aluminum nitride substrates.

The strongest change of the wetting angle could be seen with the oxygen-based substrates, fused silica and sapphire. It has to be noted that these changes are quite slow, i.e. they might not be readily noticeable when the total measurement time is only a few hours. Fig. 2a shows an example of two pictures with a time period of 5 days in between and the corresponding plot for this experiment. It is clearly visible that the wetting angle starts at a high value of around 155° and decreases over 2.5 days to an equilibrium angle of around 115°. This could be explained by a slow

oxidation process, similar to the decrease of the wetting angle with increasing oxygen partial pressure shown by many other metals [14]. Measurements of Ge–Si melts with different Si concentrations showed a similar decrease of the wetting angle, but in a much shorter time due to the higher reactivity of Si [12]. Another phenomenon noticeable with oxygen-based substrates was the change of the wetting angle with respect to the atmosphere inside the ampoule, shown in Fig. 2b. This experiment was started in dynamic vacuum until the equilibrium angle of 117° was reached. Then the atmosphere was changed to a slight overpressure of argon (1040 mbar). As shown in Fig. 2b, the wetting angle increases until it reaches a value of about 40° above the equilibrium wetting angle under vacuum (inverted triangles in Fig. 2b). Evacuating the ampoule until the equilibrium angle of 115–117° was reached again reversed this change. The second change to 1040 mbar argon led to the same increase of the wetting angle (triangles in Fig. 2b). Since one would expect that a higher oxygen partial pressure is present under argon, the simple explanation of higher oxygen partial pressure resulting in lower wetting angles might not be true for the Ge–SiO₂ system. After a certain amount of time a layer was noticeable on the surface of the drop. This is an indication that the germanium reacted with the SiO₂ substrate and/or the surrounding atmosphere. When the sample was returned to dynamic vacuum, the layer evaporated and the wetting angle reached the equilibrium angle after 62 h. Glow discharge mass spectroscopy (GDMS) measurements of the Ge drop showed high oxygen and silicon contents of up to 10³ ppm (see Table 3), pointing to a reaction with the substrate. This effect shows the importance of taking *slow* reactions and impurities into account when discussing wetting angles (and surface tension) with respect to crystal growth, where typical contact times are of the order of several hours to days.

The results for sessile drops on carbon-based substrates showed stable wetting angles, between 157° and 173°, except for the CVD diamond with 146° (Table 2). The SiC result is very close to the one for graphite. SiC is supposed to have an oxidized surface at low temperatures, and a

Table 3

Glow discharge mass spectroscopy (GDMS) measurements performed on Ge samples on different substrate materials after processing. All other elements show no increase in concentration

Sample	Substrate	Processing atmosphere	Processing time (h)	B (ppb)	C (ppb)	N (ppb)	O (ppb)	Si (ppb)
Ge	Fused silica	Dynamic vacuum/argon	9	<4	1.4×10^3	2.5×10^2	1×10^4	30
Ge	Fused silica	Dynamic vacuum/argon	144	<5	2.1×10^3	1.6×10^2	2×10^6	2×10^6
Ge	Sapphire	Dynamic vacuum/argon	245	<5	2×10^3	7.9×10^2	8.5×10^4	1.5×10^4
Ge	Glassy carbon	Dynamic vacuum/argon	8	<4	3×10^3	1.2×10^2	5×10^3	1.6×10^4
Ge	pBN	Dynamic vacuum/argon	96	<2	1×10^3	4.5×10^2	7.5×10^3	4

graphitized surface at temperatures above 1050°C under high vacuum [14]. It is thus likely that the results obtained represent a graphitized surface. Carbon-based materials are therefore possible crucible materials for detached growth, although glassy carbon appears to be less favorable due to the possible contamination mentioned below in the surface tension section.

The last group of substrates is nitrogen-based. Table 2 indicates that the wetting angle measured on Si_3N_4 and AlN substrates changes over time due to reactions with the substrate materials. In addition to a direct reaction, one has to take into account that both ceramic AlN and Si_3N_4 contain several percent of additives, e.g. Y_2O_3 , Al_2O_3 , or MgO. Furthermore, the surface chemistry of both materials is strongly determined by oxygen [14], e.g. Si_3N_4 forms layers of $\text{Si}_2\text{N}_2\text{O}$ already at room temperature that are not easily removed by heating under vacuum. In the case of pBN no changes in the wetting angle were detected. In addition to being stable and repeatable, the values measured on pBN also show quite high values of around 170°. GDMS measurements showed no significant amounts of boron in the Ge samples processed on pBN substrates (Table 3).

3.2. Surface tension

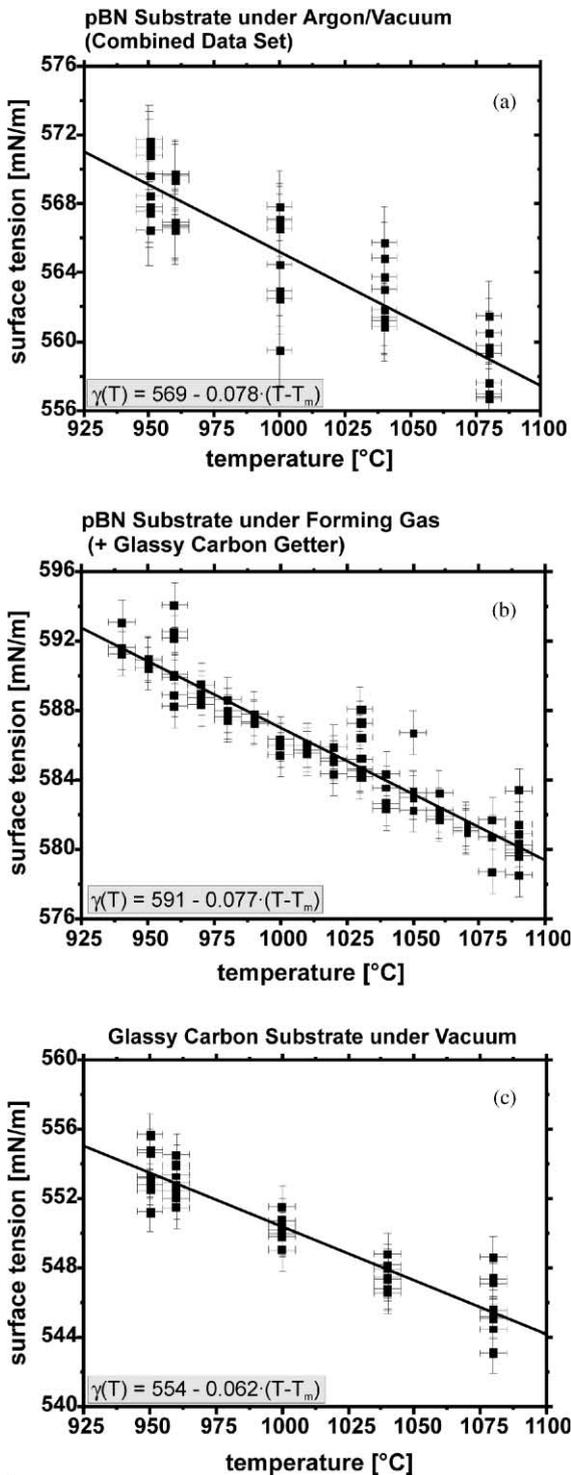
The surface tension values were also not completely independent of the substrate material, pointing to a reaction between the melt and substrate in some cases. The strongest influence on the surface tension was measured for ceramic Si_3N_4 , which showed a considerable change over time. The surface tension value for Ge on ceramic Si_3N_4 (containing 10% ceramic binder, mainly

MgO) started around 580 mN/m, and decreased to a value of about 520 mN/m within 5 h under dynamic vacuum. In the following, only those measurements without a significant change of the surface tension over time are considered; these are essentially the results on pBN and graphite substrates.

Fig. 3a shows results measured on a pBN substrate under argon and vacuum. The temperature dependence of the surface tension can be expressed as $\gamma(T) = 569 - 0.078(T - T_m)$ [10^{-3} N/m], with T_m as the melting temperature. The statistical error for Ge on pBN measured under argon and vacuum resulted in ± 2 mN/m for the absolute value² and ± 0.015 mN/m K for the temperature dependence³. The surface tension of germanium was about 3.5% higher when using forming gas and an additional carbon plate behind the substrate as oxygen getter, instead of argon with no getter, resulting in $\gamma(T) = 591 - 0.077 \times (T - T_m)$ [10^{-3} N/m] (Fig. 3b). In this case, the errors^{2,3} were ± 1.3 mN/m and ± 0.009 mN/m K, respectively. The temperature dependence is practically identical in both cases. The difference in the absolute value of the surface tension might thus be explained by the incorporation of oxygen into the melt, which could lead to a decrease in surface tension in the case without the additional oxygen getter. Measurements on graphite resulted in a similar value² of $\gamma = 585 \pm 4$ mN/m (at 955°C).

²Statistical errors as standard deviation.

³Error for the temperature dependence of the surface tension calculated by dividing the standard deviation of the surface tension value by the product of the temperature coefficient and the temperature range used in the measurements.



The results on glassy carbon need specific comment, since one would expect that they would result in similar values as with pBN or graphite. However, the experiments performed under active vacuum resulted in a surface tension value of $\gamma(T) = 554 - 0.062(T - T_m)$ [10^{-3} N/m], shown in Fig. 3c. The error was ± 1.2 mN/m for the absolute value² and ± 0.009 mN/m K for the temperature dependence³. Significantly, lower values measured on glassy carbon were corroborated by similar results for GeSi experiments [12]. The lower value of the surface tension at the melting point has to be ascribed to the incorporation of a yet unknown impurity into the melt (Fig. 4).

4. Summary and conclusions

The wetting angles and the surface tension of germanium on different substrate materials were determined using the sessile drop technique. Reactions with the substrate and incorporation of impurities into the melt lead to a decrease of the wetting angle in several cases. For samples processed on SiO₂ for several days, GDMS results showed incorporation of oxygen and silicon into the Ge samples with amounts up to 10³ ppm, see Table 3. Stable wetting angles were found for carbon-based substrates and pBN. GDMS measurements showed no significant boron incorporation in the Ge sample after processing as shown in Table 3. The highest wetting angle for Ge was found for pBN substrates with an angle of 173°.

Fig. 3. Temperature dependence of the surface tension of germanium. The vertical error bars shown are the statistical errors as standard deviation. The horizontal error bars are set to $\pm 5^\circ\text{C}$ to take thermocouple errors into account. The error for the temperature dependence of the surface tension was calculated by dividing the standard deviation of the surface tension value by the product of the temperature coefficient and the temperature range used in the measurements. Measured on pBN under 1040 mbar in (a) an argon atmosphere and under dynamic vacuum (combined data set), (b) a forming gas atmosphere with additional glassy carbon as oxygen getter (not in contact with the melt) and (c) Measured on glassy carbon under dynamic vacuum.

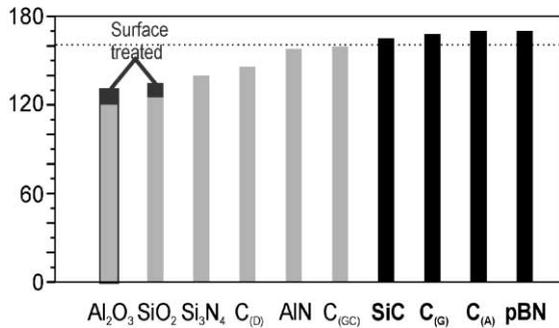


Fig. 4. Wetting angles of molten Ge for different substrates. In the case of a change due to a reaction, the stable final angle was used in the diagram. In case of the oxygen-based substrates the (higher) wetting angles measured on substrates with surface treatment (sandblasting, HF fume etching, and fire polishing) are shown for different gray values.

Table 4

Comparison of the present surface tension data with previous results. Note that the temperature dependence of the surface tension is in good agreement for all new results, i.e. [3,5,14,22] and our result. This is also true for the absolute values, except the high one measured with the ring depression technique [3]

Reference	γ (mN/m)	$\delta\gamma/\delta T$ (mN/m K)
Present results ^a	591	-0.08
[5] ^b	583	-0.08
[3] ^c	665	-0.08
[3] ^a	590	-0.07
[14,22] ^a	587	-0.105
[18]	600	-0.12
[20] ^d	616	-0.094
[21] ^d , [19]	621	-0.26
[23] ^e	600	—

^a Sessile drop method.

^b Levitating drop method.

^c Ring depression technique.

^d Maximum bubble pressure method.

^e Pendant drop.

This corroborates with the GeSi measurements [12] and the measurements for Si performed by Mukai et al. [15] where the highest contact angles were measured on BN substrates with a value of 145°. For the surface tension, impurities in the system play an important role in the absolute value. The surface tension measured on pBN

substrates under forming gas and with a glassy carbon piece as oxygen getter showed the most reliable values with $\gamma(T) = 591 - 0.077(T - T_m)$ [10^{-3} N/m]. This value is 1.5% higher than the result of Rhim et al. [5] measured with the crucible-free high-temperature electrostatic levitator (HTESL), and very close to the value of Nakanishi cited in Ref. [3]. Measurements on pBN under vacuum and argon (and without the additional glassy carbon plate) gave $\gamma(T) = 569 - 0.078(T - T_m)$ [10^{-3} N/m], 2.5% below the result of Rhim [5]. His result of $\gamma(T) = 583 - 0.08 \times (T - T_m)$ [10^{-3} N/m] shows essentially the same temperature dependence and an absolute value closer to our higher value. Nakamura et al. [16] state that oxygen has a strong effect on the surface tension of silicon melts. Mukai et al. [17] measured the dependence of the surface tension from the oxygen partial pressure P_{O_2} in the system and stated a significant decrease of the surface tension of Si with increasing P_{O_2} . They also found a slight increase of the surface tension when $P_{O_2} > P_{O_{2sat}}$ (with $P_{O_{2sat}}$ = the saturated oxygen partial pressure in the system). Therefore, we assume that a small oxygen content in our system for Ge on pBN measured without H₂ or carbon oxygen getter reduced the absolute value of the surface tension. The temperature dependence of the surface tension shows good agreement among our experiments and also with the temperature dependences measured in the last few years [3,5,14,22] (see Table 4).

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