Interfacial thermal conductance of 7075 aluminum alloy microdroplets in contact with a solid at fast melting and crystallization

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Abstract
Ultrafast nanocalorimetry, in combination with high-speed IR thermography, is used to measure the interfacial thermal conductance (ITC) of the thermal contact of metal microdroplets with a solid during fast melting (including laser heating). IR thermography and membrane nanocalorimetry were used to measure the temperature difference at the membrane/sample interface during the melting and crystallization of aluminium alloy (AA7075) microdroplets (20 μm in diameter) over a wide range of heating and cooling rates (up to 10^5 K s^{-1}). This is the first time ITC has been measured at such high heating and cooling rates with this new method. We found that the interfacial temperature difference reaches about 80 K during the solidification of microdroplets during laser heating. This result is significant for understanding various industrial laser-assisted processes. It has been established that ITC measured for AA7075 microdroplets gradually increases by an order of magnitude during melting in the range from the solidus temperature to the liquidus temperature of the alloy. This unusual behavior of ITC during melting can be important for understanding and optimizing laser-assisted additive manufacturing processes.

Nomenclature
Latin Symbols
b_0 distance from the center to the periphery of the membrane (m)
c_p specific heat of the sample (J g^{-1} K^{-1})
C_S sample heat capacity (J K^{-1})
d_m membrane thickness (μm)
g acceleration of gravity (m s^{-2})
G thermal contact conductance (W K^{-1})
G_L thermal contact conductance with liquid sample (W K^{-1})
Gr Grashof number (dimensionless)
H_S sample melting enthalpy (nJ)
h_f specific heat of fusion (J g^{-1})
IRI(t) intensity of thermal radiation emitted from the sample surface (r.u.)
L_l distance from the hot zone of the membrane to the thermostat (m)


$m_s$ sample mass (ng)
$Nu$ Nusselt number (dimensionless)
$Pr$ Prandtl number (dimensionless)
$P(t)$ heater power (W)
$P_s(t)$ power absorbed by the sample (W)
$q_{ms}$ heat flux from membrane to sample (W·m$^{-2}$)
$R(t)$ scan rate (K·s$^{-1}$)
$r_s$ sample radius ($\mu$m)
$r_h$ heater radius ($\mu$m)
$S_0$ sample cross-sectional area (m$^2$)
$S_C$ thermal contact area (m$^2$)
$S_S$ area of the sample hemisphere (m$^2$)
$t_1, t_2$ moments of the onset and the end of melting (s)
$t_3, t_4$ moments of the onset and the end of crystallization (s)
$t_{\text{max}}$ middle of the heating-cooling scan (s)
$T_m$ membrane temperature (K)
$T_S$ sample temperature (K)
$T_t$ thermostat temperature (K)
Greek Symbols
$\alpha_g$ heat loss parameter (membrane heating) (W·m$^{-2}$·K$^{-1}$)
$\alpha_{gl}$ heat loss parameter (laser heating) (W·m$^{-2}$·K$^{-1}$)
$\Delta t_c$ crystallization time (membrane heating) (s)
$\Delta t_l$ crystallization time (laser heating) (s)
$\Delta T_S$ temperature difference across the sample (K)
$\Delta T_{ms}$ temperature difference across the membrane/sample interface (K)
$\varepsilon$ membrane emissivity (dimensionless)
$\lambda$ thermal conductivity of sample (W·K$^{-1}$·m$^{-1}$)
$\lambda_g$ thermal conductivity of gas (W·K$^{-1}$·m$^{-1}$)
$\nu_g$ kinematic viscosity of gas (m$^2$·s$^{-1}$)
$\rho_s, \rho_g$ density of sample and gas (kg·m$^{-3}$)
$\sigma$ Stefan-Boltzmann constant (W·m$^{-2}$·K$^{-4}$)

1. Introduction

Fast melting and solidification of metal microdroplets are very important in many industrial processes, for example, in laser welding \cite{1} and laser-assisted additive manufacturing \cite{2}. Heat transfer between a metal microdroplet and a substrate is highly dependent on changes in interfacial thermal conductance (ITC). Thus, quantifying ITC is important for optimizing melting and solidification processes in applications such as laser additive manufacturing. Computer numerical simulations is one of the methods to understand the processes during additive manufacturing and their influence on the final product properties. Finite elements analysis, complex flow dynamics and even machine learning are applied \cite{3–5}. However, the efforts are hampered by the difficulties of direct experimental observations. Particularly, modeling of powder particle interactions requires experimental data on surface heat transfer during the laser melting process \cite{6}.

ITC affects the rate of solidification, and the rate of solidification affects the development of the microstructure and mechanical properties of the resulting material \cite{1, 7–13}. Local cooling rates during solidification in laser additive manufacturing can be thousands or even millions K·s$^{-1}$ \cite{2, 7, 14}. Ultrafast nanocalorimetry makes it possible to measure the rapid melting and solidification of micro-sized samples at rates up to millions of K·s$^{-1}$ and even higher \cite{15–18}. The measured microdroplet is commonly heated by a thin film heater located on the calorimeter membrane (membrane heating). However, the microdroplet can
alternatively be heated by an external diode laser (laser heating) [19]. Our goal is to measure interfacial heat transfer between metal microdroplets and a substrate during fast melting and solidification at a temperature scan rate in the range from $10^3$ K s$^{-1}$ to $10^5$ K s$^{-1}$. The sample temperature was scanned in two modes with membrane and laser heating. The advantage of membrane heating is the ability to accurately measure the power absorbed (released) by the sample during melting (crystallization). The advantage of laser heating is the possibility of local heating of the microdroplet, similar to laser additive manufacturing, with the possibility of local measurement of the temperature of the microdroplet at the same time. The disadvantage of laser heating is that, so far, it is not possible to determine the power absorbed by the sample accurately. However, the possibility of carrying out measurements in both modes makes it possible to obtain reliable information on the ITC between the measured microdroplets and the substrate during fast melting and solidification. The melting and solidification of aluminium alloy microdroplets (AA7075) with a diameter of about 20 μm was studied; such microparticles are used in additive manufacturing. AA7075 is almost as strong as steel but weighs about 3 times less, so this material is often used in industry.

In our previous article [20], it was shown for pure tin microdroplets that a sharp jump in the interfacial thermal conductance $G(T)$ occurs immediately by an order of magnitude when the sample surface becomes liquid just before the melting point. A jump in $G(T)$ just before the melting point was observed in experiments with pure aluminum, copper, and stainless steel [21]. However, an unusual gradual increase in $G(T)$ with temperature was recently discovered for a microdroplet of pure indium in the temperature range of premelting [22]. The gradual change in $G(T)$, found for pure indium microdroplets, indicates a gradual change in the strength of the interaction between the sample and the substrate during the melting of the sample. A similar gradual change in $G(T)$ during the melting of AA7075 microdroplets in contact with a solid substrate was observed in this article.

The advantage of the method used in this work is the combination of ultrafast nanocalorimetry with high-speed IR thermography. Thus, using this new method, it is possible to measure ITC at high heating and cooling rates up to $10^5$ K s$^{-1}$, which makes it possible to simulate melting and crystallization processes occurring in laser-assisted technologies. The proposed method of ITC determination particularly represents the process of the laser powder bed fusion. In the case of additive manufacturing, the spherical particle thermal contact is complicated by the particle shape, surface defects, presence and thickness of an oxide layer and surface modifications with e.g. nucleating agents. All these parameters can be reproduced by the combined FSC IR experiments.

In the first part of the article, we discuss the experimental method and measurements of the parameters of AA7075 microdroplets. Then, measurements of ITC during fast melting and crystallization are presented for several temperature scan rates in laser and membrane heating modes.

2. Ultrafast nanocalorimetry combined with IR thermography and laser heating

2.1. Calorimetric sensor and nanocalorimetry

The membrane calorimetric sensor (XEN-39469 from Xensor Integration, NL) was used for ultrafast nanocalorimetry of metal microdroplets during their melting and crystallization. The sensor consists of a thin (about 1 μm) amorphous silicon nitride membrane with a resistance heater and thermocouples located at the central (hot zone) of the membrane, see figure 1. Thermocouples, heater, and electrical connections are formed by p-type and n-type doped polysilicon tracks. The central zone of the membrane is coated with a submicron layer of gold to smooth out temperature gradients in this zone. The electrical circuit is protected by a submicron layer of amorphous silicon nitride. Thus, the total membrane thickness $d_m$ is about 1.5 μm. The measured sample with a radius of $r_0 = 10$ μm is placed on the hot junctions of two thermocouples in the center of the membrane using an optical microscope, see figure 1. To show thermocouples, the sample is off-center.

However, the measured sample was located in the center, see figure 2(a). During measurements after the first melting, the position of the sample and its shape remain stable due to the cohesive forces acting between the membrane and the sample.

The sample was prepared under a binocular microscope with a variable 10x–100x magnification. Powder particles were preselected and placed onto a G300F1 TEM grid finder (purchased from Science Services GmbH, Germany) for further selection by size. Several 10 to 20 μm in diameter particles without satellites or defects were selected for the aimed temperature scanning rate range up to $10^5$ K s$^{-1}$. After that, a single particle was moved on the sensor surface, on the side protected by a thin SiN layer, to avoid alloying with gold elements of the system, see figure 1. The sample was located just above the thermopiles to ensure the lowest measurement lag and fastest temperature control system response. The fast scanning calorimetry equipment is described in detail in [17]. More information about the equipment for IR thermography is presented in [23–26].
The central hot zone of the membrane is heated by a heater consisting of two concentric polysilicon tracks, the radius of the outer track is $r_h = 75 \mu m$. The distances $b_0$ and $L_t$ from the center of the membrane to the silicon frame are about 400 $\mu m$ and 300 $\mu m$, respectively, see figure 1.

The sensor is installed in a thermostat with regulated temperature $T_t$ and stable ambient gas pressure. The ambient gas (air) provides heat exchange between the central hot zone of the membrane and the thermostat. The heat loss from the central hot zone due to the thermal conductivity of the gas $\lambda_g$ is proportional to $\lambda_g (T - T_t) / L_t$ at $L_t \ll \eta_t$. In the opposite case, the heat loss is proportional to $\lambda_g (T - T_t) / \eta_t$ at $L_t \gg \eta_t$, see appendix. The size of the central hot zone $r_h$ is small with respect to $L_t$. The membrane temperature $T_m(t, r)$ decreases exponentially with distance $r$ from the center [18]. Thus, the heat generated by the heater is removed by the ambient gas before it reaches the periphery of the membrane; heat transfer between the membrane and the frame at the periphery of the membrane is negligible [18]. Thus, the heat loss from the central hot zone of the membrane is proportional to $\lambda_g (T - T_t) / \eta_t$. The contribution of thermal radiation and convection to heat loss is relatively small at micrometer sizes; these contributions are calculated in appendix.

The heat capacity $C_S$ of the sample can be determined from equation (1)

$$(C_S + C_A) \frac{\partial T}{\partial t} = P(t) - B(T - T_t)$$

where $C_A$ is the addenda heat capacity, $P(t)$ is the power of the heater located on the membrane, $T$ is the temperature of the sample measured by the thermocouples, and the factor $B$ describes the heat transfer from the heating zone to the thermostat. Two identical membrane sensors are installed on the standard TO5 housing, which allows the use of a differential power compensation scheme [17]. A power compensated analog differential control loop accurately controls the sample temperature [17], allowing measurements to be made at a constant
scan rate $R$ in the range of $10^3 \text{K s}^{-1} \leq R \leq 10^5 \text{K s}^{-1}$. The time resolution of calorimetric measurements varies from 6 μs to 52 μs at different scan rates $R$. Temperature resolution of about ±0.5 K was determined by random noise at $10^3 \text{K s}^{-1} \leq R \leq 2 \times 10^4 \text{K s}^{-1}$. However, a temperature resolution of 1.2 K was associated with a time resolution of 12 μs at $R = 10^5 \text{K s}^{-1}$.

2.2. Laser heating

The measurements were carried out in two modes, when the sample was heated by the heater located on the membrane (membrane heating) and by an external diode laser (laser heating). The laser spot diameter of about 8 μm was significantly smaller than the sample size. Thus, heating with a finely focused diode laser (830 nm) makes it possible to apply energy directly to the sample, avoiding heating the membrane near the sample. Positioning of the laser spot on the sample is possible with a high-resolution IR camera, see below. The power of the diode laser can be precisely adjusted from zero to about 100 mW. Thus, the power compensation scheme makes it possible to control the sample temperature during laser heating in the same way as during membrane heating [19]. In the case of laser heating, we have gained the advantage of local heating with simultaneous local temperature measurement. The disadvantage of laser heating was that, in this case, it was not possible to determine the power absorbed by the sample accurately.

In the case of laser heating of the sample, the radius of the hot zone is now equal to $r_s$. Thus, the heat loss is proportional to $λ_2 / r_s$, see appendix. An IR image of the central zone of the membrane with the sample during laser heating is shown in figure 2(a). It should be noted that the emissivity of a metal surface is much lower than that of the polysilicon tracks. The emissivity of materials with low electrical resistivity is less than that of materials with higher electrical resistivity [27, 28]. Therefore, the radiation intensity of doped polysilicon tracks is much higher than that of metal surfaces. In addition, the emissivity of the transparent membrane is much lower than that of the other surfaces, figure 2.

Denote by $T_m(t)$ and $T_b(t)$ the temperature of the membrane and the sample at the membrane/sample interface. The interfacial temperature difference $\Delta T_{mb}(t) = T_m(t) - T_b(t)$ depends on the ITC between the sample and the membrane. For further discussion, we are interested in the temperature difference $\Delta T_{mb}(t)$ at the membrane/sample interface.

2.3. IR thermography

To measure the interfacial temperature difference $\Delta T_{mb}(t)$, it is necessary to measure the temperature on both sides of the interface, which is a complicated task for a microdroplet during fast melting and crystallization. This problem can be solved using non-contact IR thermography combined with nanocalorimetry [20, 22]. The temperature of the sample surface on the side opposite the membrane can be calculated from the intensity of thermal radiation $IRI(t)$ emitted from the sample surface. This temperature is close to the temperature at the membrane/sample interface with an error of about the temperature difference $\Delta T_m$ across the sample; this temperature difference is negligible due to the high thermal conductivity and very small size of the sample. However, the temperature difference at the membrane/sample interface is significant and reaches about 80 K in laser heating experiments, see below.

In this work, we used a high-speed IR camera with an array of InSb photodetectors and a special Si-Ge microscopic lens [23–26]. The system operates in the wavelength range of about 3–5 μm and has a spatial resolution of 4 μm. Thermal images of the IR camera are taken at an exposure time of 0.2 ms and a sampling rate of 3800 Hz, i.e. time resolution of 0.26 ms. The measured time dependences of $IRI(t)$ and $T_m(t)$ (together with $P(t)$) was synchronized with an accuracy of about 0.2 ms using a very sharp onset of crystallization, see the next section.

3. Results

3.1. IR thermography

The intensity $IRI(T_b)$ of thermal radiation, measured by the IR camera at sample temperature $T_b$, can be represented by the following equation: $IRI(T_b) = AT_b^4 + IRI_\text{bg}$, where the factor $A$ depends on the emissivity of the sample surface, and the background level $IRI_\text{bg}$ depends on the thermal noise and dark current of photodetectors [18]. The parameters $A$ and $IRI_\text{bg}$ can be excluded if any two reference points are known, say, temperatures $T_a$ and $T_b$ are known, at which $IRI(T_a)$ and $IRI(T_b)$ are measured. The sample temperature $T_S$ can then be calculated from the measured $IRI(T_b)$ according to equation (2).

$$T_S = T_a(1 + ((T_b / T_a)^4 - 1)(IRI(T_b) - IRI(T_a)) / (IRI(T_b) - IRI(T_a)))^{1/4}$$

The membrane temperature $T_m(t)$ was linearly scanned from 600 K to the maximum $T_m(t_{\text{max}}) = 1050$ K and back to 600 K, where $t_{\text{max}}$ is the middle of the heating-cooling scan. Thus, the membrane temperature is equal to
the maximum value $T_m^{\text{max}} = 1050$ K and rate $R$ is equal to zero at $t_{\text{max}}$ see figure 3. Denote by $t_1$, $t_2$, $t_3$, and $t_4$ the moments of the onset of sample melting, the end of melting, the onset of crystallization, and the middle of crystallization, respectively, see figures 3–5. Unfortunately, the onset of melting of the alloy is indistinct and cannot be accurately determined. However, the end of the sample melting can be clearly indicated, see figures 3 and 4. The value of the radiation intensity at the end of melting ($t = t_2$) is fully reproducible. For example, at $R = 1000$ K s$^{-1}$, this intensity $t_{\text{IRI}}(t_2) = (14140 \pm 20)$ r.u. corresponds to the liquidus temperature of the alloy, see figure 3. The liquidus temperature of AA7075 is equal to $T_{\text{Liq}} = 901$ K [29, 30]. Thus, the point $IRI(t_{\text{IRI}}) = 14140$ r.u. can be used for temperature calibration of measurements of the intensity of thermal radiation. In addition, the point $IRI(t_{\text{max}}) = 15580$ r.u at zero rate $R$ corresponds to the sample temperature $T(t_{\text{max}})$, which is close to $T_m^{\text{max}} = 1050$ K. More precisely, $T_s^{\text{max}} = 1042$ K can be obtained from $T_m^{\text{max}}$ as described in the Appendix. Thus, the value of the sample temperature $T(t_{\text{max}})$ can be obtained from the measured $IRI(t)$ according to equation (2), where $T_s = T_{\text{Liq}}$ and $T_b = T_s^{\text{max}}$.

Similar time dependencies of the intensity $IRI(t)$, temperature $T_m(t)$, and power $P(t)$ were measured at different scan rates. For example, at $R = 5000$ K s$^{-1}$, see figure 6.

Thus, we obtain the sample temperature $T(t)$ at specific points in the scanning process, which can be used to calculate the thermal contact conductance at the membrane/sample interface. For example, the results at $R = 1000$ K s$^{-1}$ and 5000 K s$^{-1}$ are collected in tables 1 and 2. Note that the membrane temperature in the middle of the crystallization process (at time $t_3$) is approximately equal to $T_m(t_3)$, see figure 5. Similar results were obtained at various $R$ in the range of $10^3–10^5$ K s$^{-1}$.

Similar time dependencies of the intensity $IRI(t)$ and temperature $T_m(t)$ were measured at laser heating at different $R$, for example, at $R = 1000$ K s$^{-1}$ and 5000 K s$^{-1}$, see figures 7 and 8. The membrane temperature $T_m(t)$ was linearly scanned from 500 K to the maximum $T_m(t_{\text{max}}) = 1000$ K and back to 500 K.
Thus, at laser heating, we obtain the sample temperature $T_s(t)$ at specific points in the scanning process, which can be used to calculate the thermal contact conductance at the membrane/sample interface. For example, the results at $R = 1000 \text{ K s}^{-1}$ and $5000 \text{ K s}^{-1}$ are collected in tables 3 and 4. In the case of laser heating,
Figure 7. Time dependence of the intensity $I(t)$ and membrane temperature $T_{mm}(t)$ during heating-cooling scan (a) and crystallization (b) at laser heating ($R = 1000 \text{ K s}^{-1}$).

Figure 8. Time dependence of the intensity $I(t)$ and membrane temperature $T_{mm}(t)$ during heating-cooling scan (a) and crystallization (b) at laser heating ($R = 5000 \text{ K s}^{-1}$).

| Time in s | $T_{mm}(t)$ in K | $I(t)$ in r.u. | $T_{TS}(t)$ in K |
|-----------|------------------|----------------|------------------|
| $t_2$     | 848              | 13330          | 901              |
| $t_{max}$ | 1000             | 15300          | 1000             |
| $t_3$     | 803.4            | 12840          | 870.1            |
| $t_4$     | 813.5            | 13180          | 891.9            |

Table 3. Parameters of the scan process at specific points in time at $R = 1000 \text{ K s}^{-1}$ and laser heating.

| Time in s | $T_{mm}(t)$ in K | $I(t)$ in r.u. | $T_{TS}(t)$ in K |
|-----------|------------------|----------------|------------------|
| $t_2$     | 862              | 13410          | 901              |
| $t_{max}$ | 1000             | 15300          | 1000             |
| $t_3$     | 805.5            | 12920          | 868.7            |
| $t_4$     | 816              | 13230          | 889.6            |

Table 4. Parameters of the scan process at specific points in time at $R = 5000 \text{ K s}^{-1}$ and laser heating.
we assume that $T_S(t_{\text{max}})$ is approximately equal to $T_m(t_{\text{max}})$. Similar results were obtained at various $R$ in the range of $10^3$–$10^5$ K s$^{-1}$.

Thus, the temperature $T_S(t)$ of the sample and the temperature $T_m(t)$ of the membrane can be measured simultaneously. It is then possible to measure the change in the interfacial temperature difference $\Delta T_{mS}(t)$ during fast melting and crystallization of metal microdroplets.

### 3.2. Calorimetry

The mass of the sample can be determined from the measured enthalpy $H_S$ of melting and crystallization of the sample. The measurements were carried out at scan rate $R$ in the range of $10^3$–$10^5$ K s$^{-1}$. The time dependences of the membrane temperature $T_m(t)$ and power $P(t)$ during melting and crystallization of the sample at $R = 10^3$ K s$^{-1}$ are shown in figures 4 and 9, as well as in figures 10 and 11 at $R = 5 \cdot 10^3$ K s$^{-1}$. The power absorbed (released) by the sample during melting (crystallization) is equal to $P(t) = P(t) - BL(t)$, where $BL(t)$ is the baseline, see figures 4, 9, 10, and 11. Thus, the enthalpy $H_S$ of melting (crystallization) of the sample is the area under the curve $P(t) - BL(t)$. It should be noted that after the completion of sample melting, the membrane temperature $T_m(t)$ becomes overheated for a short time, see figures 4(a) and 10(a). This overheating is compensated by the power control undershoot. The extra heat absorbed by the membrane after the completion of sample melting is compensated by a further undershoot in power $P(t)$. Thus, the total area under the curve $P(t) - BL(t)$ in time intervals with a positive and negative difference $P(t) - BL(t)$ is equal to the heat absorbed by the sample during melting. The same applies to the crystallization of the sample, see figures 9 and 11.
The average value of $H_S$ measured during melting and crystallization at different scan rates is equal to $H_S = 4.0 \pm 0.4$ $\mu$J. The uncertainty of baseline is responsible for the 10% error in the measured $H_S$ value. The sample mass $m_S = 11.2 \pm 1$ ng is obtained from $H_S$ and the specific heat of fusion $\Delta h_f = 358$ J g$^{-1}$ [29, 30]. The thermophysical properties of the material AA7075 are available in [29, 30] and are summarized in table 5.

Thus, the heat capacity of the sample is equal to $C_p = 12.6 \pm 1$ nJ K$^{-1}$ at $T \geq T_{leq}$. The sample radius is equal to $r_S = 10.2 \pm 1$ $\mu$m at $T \geq T_{leq}$ and $\rho = 2.5$ $\mu$m, since the shape of the sample is close to spherical. The areas of the hemisphere and the cross section of the sample are $S_h = 2\pi r_S^2$ and $S_0 = \pi r_S^2$, respectively. Sample parameters are collected in table 6.

### 4. Discussion

#### 4.1. Interfacial thermal conductance at membrane heating

The interfacial temperature difference $T_m(t) - T_S(t)$ is proportional to the heat flow $q_{ms}$ across the membrane/sample interface and is inversely proportional to the thermal contact area $S_C$ and the ITC, measured in W m$^{-2}$K$^{-1}$. Consider the product of this thermal conductance and the contact area $S_C$. Let $G$ denote this product, measured in W/K. The thermal contact area $S_C$ cannot be accurately measured in this experiment. Therefore, we consider the interfacial thermal conductance $G$, measured in W/K, which can be determined from equation (3).

$$q_{ms} = (T_m - T_S)G,$$

To determine the sample temperature $T_S(t)$ at the membrane/sample interface, we neglect the temperature difference $\Delta T_S$ across the sample and use the sample temperature determined from the intensity $IRI(t)$ of thermal radiation emitted from the sample surface opposite the membrane. $\Delta T_S \leq q_{\text{max}}r_S/\lambda S_0$, where $q_{\text{max}}$ is the maximum heat flow through the sample. This heat flow is maximum at crystallization: $q_{\text{max}} = H_S/\Delta t_{cr}$,
where the crystallization time $\Delta t_{cr}$ is about 1.8 ms, see figure 5. Thus, $q_{\text{max}} = 2.2$ mW at $\Delta t_{cr} = 1.8$ ms and $H_{l} = 4.0 \mu$J, see table 6. Therefore, $\Delta T_{S} \leq 1$ K at $\lambda = 85$ W m$^{-1}$ K$^{-1}$ for liquid sample, see table 5.

Let $G_{l}$ denote the interfacial thermal conductance at the interface between the sample and the membrane for a molten (liquid) sample. Let us consider $G_{l}$ during melting and crystallization of the measured microdroplet.

Interfacial thermal conductance $G_{l}$ at the end of the melting process (at $t_{2}$) can be calculated from the energy balance equation (4), where $P_{l}(t_{2})$ is the power absorbed by the sample at $t_{2}$. The power $G_{l}(T_{m} - T_{S})$ supplied from the membrane to the sample is spent on melting the sample, heat loss from the sample to the surrounding gas $\alpha_{g} (T_{S} - T_{l})S_{l}$, and heating the sample $C_{S}R$ at a rate $R$. The thermostat temperature in all experiments was equal to $T_{l} = 300$ K. Therefore,

$$G_{l} = (P_{l}(t_{2}) + \alpha_{g}(T_{S}(t_{2}) - T_{l})S_{l} + C_{S}R)/(T_{m}(t_{2}) - T_{S}(t_{2}))$$

Thus, at $R = 1000$ K s$^{-1}$, $P_{l}(t_{2}) = 0.3$ mW (see figure 4), $T_{m}(t_{2}) = 922$ K, $T_{S}(t_{2}) = 901$ K (see table 1), $\alpha_{g}(T_{S}(t_{2}) - T_{l})S_{l} = 0.37$ mW at $\alpha_{g} = 928$ W m$^{-2}$ K$^{-1}$ (see appendix), and $C_{S}R$ is negligible at $C_{S} = 12.6$ mJ K$^{-1}$ (see table 6). Thus, $T_{m}(t_{2}) - T_{S}(t_{2}) = 21$ K and $G_{l} = 4.6 \times 10^{-3}$ W K$^{-1}$.

Correspondingly, at $R = 5000$ K s$^{-1}$, $P_{l}(t_{2}) = 1.1$ mW (see figure 10), $T_{m}(t_{2}) = 930$ K, $T_{S}(t_{2}) = 901$ K (see table 2), $\alpha_{g}(T_{S}(t_{2}) - T_{l})S_{l} = 0.37$ mW, and $C_{S}R$ is negligible. Thus, $T_{m}(t_{2}) - T_{S}(t_{2}) = 29$ K and $G_{l} = 5.1 \times 10^{-5}$ W K$^{-1}$. Similar results are obtained at different rates $R$, see table 7. The uncertainty of this result is about 50% due to the following main sources of error. The uncertainty of the interfacial temperature difference $\Delta T_{mS}$ is about 30%. This uncertainty is related to the $T_{l}$ measurement error (about 7 K) obtained from the IRI measurements, see appendix. The uncertainty of the heat losses is about 30%, see Appendix. The error in measuring $P_{l}$ during melting is about 30% due to the baseline error.

In addition, the interfacial thermal conductance $G_{l}$ can be calculated from the energy balance equation averaged over the crystallization time interval $\Delta t_{cr}$ is 1.8 ms, see equation (5). The energy $H_{l} = 4.0 \mu$J released by the sample during crystallization is spent on heat loss from the sample to the surrounding gas $\alpha_{g}(T_{S} - T_{l})S_{l} \Delta t_{cr}$, heat transferred from the sample to the membrane $G_{l}(T_{S} - T_{m}) \Delta t_{cr}$, and heating the sample from $T_{S}(t_{3})$ to $T_{l}(t_{4})$. Therefore,

$$G_{l} = (H_{l} - \alpha_{g}(T_{S}(t_{4}) - T_{l})S_{l} \Delta t_{cr} - C_{S}(T_{S}(t_{4}) - T_{l}(t_{3}))) / \Delta t_{cr}$$

Thus, at $R = 1000$ K s$^{-1}$, $\alpha_{g}(T_{S}(t_{4}) - T_{l})S_{l} \Delta t_{cr} = 0.64 \mu$J, $C_{S}(T_{S}(t_{4}) - T_{l}(t_{3})) = 0.75 \mu$J, $T_{S}(t_{3}) = 887.1$ K, $T_{l}(t_{3}) = 827.6$ K, and $T_{m}(t_{4}) = 859$ K (see table 1). Thus, $T_{m}(t_{4}) - T_{S}(t_{3}) = 28.1$ K and $G_{l} = 5.2 \times 10^{-5}$ W K$^{-1}$.

Correspondingly, at $R = 5000$ K s$^{-1}$, $\alpha_{g}(T_{S}(t_{4}) - T_{l})S_{l} \Delta t_{cr} = 0.63 \mu$J, $C_{S}(T_{S}(t_{4}) - T_{l}(t_{3})) = 0.85 \mu$J, $T_{S}(t_{3}) = 872.6$ K, $T_{l}(t_{3}) = 805.5$ K, and $T_{m}(t_{4}) = 848.5$ K (see table 2). Thus, $T_{m}(t_{4}) - T_{S}(t_{3}) = 24.1$ K and $G_{l} = 5.8 \times 10^{-5}$ W K$^{-1}$. Similar results are obtained at different rates, see table 7. The uncertainty of this result is about 50% due to the following main sources of error. The uncertainty of the interfacial temperature difference $\Delta T_{mS}$ is about 30%. The uncertainty of the numerator of the fraction on the right side of equation (5) is about 20%. The error of the time interval $\Delta t_{cr}$ is about 30% due to the time resolution of the IR camera of about 0.26 ms.

### 4.2. Interfacial thermal conductance at laser heating

In the case of the laser heating of the sample, the radius of the hot zone is equal to $r_{l}$. Thus, the heat loss is proportional to $\lambda_{l}/r_{l}$ and the parameter of the heat loss from the sample surface is equal to $\alpha_{gl} = 6.149$ W m$^{-2}$ K$^{-1}$, see appendix. With laser heating, the crystallization time $\Delta t_{l}$ is about 0.5 ms, see figures 7 and 8. Therefore, the maximum heat flow $q_{\text{max}}$ through the sample is equal to $H_{l}/\Delta t_{l} = 8$ mW. The temperature difference $\Delta T_{S}$ across the sample is $\Delta T_{S} \leq q_{\text{max}}r_{l}/\lambda_{l}S_{l}$. Thus, $\Delta T_{S} \leq 3$ K at $\lambda = 85$ W m$^{-1}$ K$^{-1}$ for the liquid sample, see table 5.
With laser heating, it was impossible to determine the power absorbed by the sample accurately. Thus, it was not possible to measure the interfacial conductance \( G_t \) during melting. However, \( G_t \) can be obtained from the energy balance equation (6) during crystallization, since the energy \( H_S \) released during crystallization is known, see table 6. Therefore, similarly to equation (5), we have

\[
G_t = \frac{(H_S - \alpha_g(T_s(t_1) - T_s)S_G\Delta t_t - C_S(T_s(t_2) - T_s(t_1))) / \Delta t_t (T_s(t_1) - T_m(t_2))}{\Delta t_t (T_s(t_1) - T_m(t_2))}
\]

Thus, at \( R = 1000 \text{ K s}^{-1} \), \( \alpha_g(T_s(t_1) - T_s)S_G\Delta t_t = 1.19 \mu \text{J} \), \( C_S(T_s(t_2) - T_s(t_1)) = 0.27 \mu \text{J} \), \( T_s(t_2) = 870.1 \text{ K}, \) and \( T_m(t_4) = 813.5 \text{ K} \) (see table 3). Thus, \( (T_s(t_1) - T_m(t_2)) = 78.4 \text{ K} \) and \( G_t = 6.5 \times 10^{-5} \text{ W K}^{-1} \).

Correspondingly, at \( R = 5000 \text{ K s}^{-1} \), \( \alpha_g(T_s(t_1) - T_s)S_G\Delta t_t = 1.19 \mu \text{J} \), \( C_S(T_s(t_2) - T_s(t_1)) = 0.26 \mu \text{J} \), \( T_s(t_2) = 889.6 \text{ K}, \) \( T_s(t_2) = 868.7 \text{ K}, \) and \( T_m(t_4) = 816 \text{ K} \) (see table 4). Thus, \( (T_s(t_1) - T_m(t_2)) = 73.6 \text{ K} \) and \( G_t = 6.9 \times 10^{-5} \text{ W K}^{-1} \).

The uncertainty at laser heating is about 60% due to the following main sources of error. The interfacial thermal conductance \( G_t \) obtained for the process of melting and crystallization under different heating modes and different rates, we get \( G_t = (6.0 \pm 3) \times 10^{-5} \text{ W K}^{-1} \). The measurement error of about 50% is due to the following components. The error of the interfacial temperature difference \( \Delta T_{anS} \) is about 10% and 30% at laser and membrane heating, respectively. This uncertainty is due to the \( T_s \) measurement error obtained from the IR measurements. The uncertainty of the time interval \( \Delta t_t \) and \( \Delta t_r \) is about 50% and 30% for laser and membrane heating, respectively. This error is related to the time resolution of the IR camera. The uncertainty of heat loss is about 30%. The error in measuring the enthalpy \( H_S \) during crystallization and the power \( P_s \) during melting is about 10% and 30%, respectively. This error is due to baseline uncertainty.

The interfacial thermal conductance \( G_t / S_C \) (in \( \text{W m}^{-2}\text{K}^{-1} \)) is greater than \( G_t / S_0 = 1.8 \times 10^5 \text{ W m}^{-2}\text{K}^{-1} \) since the contact area \( S_C \) is at least smaller than the cross-sectional area \( S_0 = 3.3 \times 10^{-10} \text{ m}^2 \), see table 6. The ITC can vary in a wide range of \( 10^3 \text{ to} 10^8 \text{ W m}^{-2}\text{K}^{-1} \) depending on the adhesion between the sample and the substrate [20, 22, 31–33]. The ITC measured in this article is consistent with the literature data. However, this is greater than the ITC measured for other metal microdroplets. For example, the ITC of microdroplets of liquid tin and indium is about \( 10^4 \text{ W m}^{-2}\text{K}^{-1} \) and \( 5 \times 10^4 \text{ W m}^{-2}\text{K}^{-1} \), respectively [20, 22]. This result indicates that aluminum alloy microdroplets (AA7075) in the molten state have excellent adhesion to the substrate, even better than pure tin and indium microdroplets.

### 4.3. Variation of the interfacial thermal conductance during sample melting

The variation of the interfacial thermal conductance \( G(t) \) during the melting process can be calculated from the energy balance equation (7), similar to equation (4).

\[
G(t) = (P_3(t) + \alpha_g(T_s(t) - T_s)S_G + C_S R(t)) / (T_m(t) - T_s(t))
\]

where \( P_3(t) \) is the power absorbed by the sample during melting (for example, see figure 4 at \( R = 1000 \text{ K s}^{-1} \)). \( T_s(t) \) is calculated from \( IRI(t) \) according to equation (2). Thus, we obtain the temperature difference \( T_m(t) - T_s(t) \) and the interfacial thermal conductance \( G(t) \) during the melting process. As a result, the dependence of the interfacial thermal conductance \( G \) on the temperature of the sample in the melting range is obtained, see figure 12. Note that figure 12 is a plot of the ITC versus the sample temperature calculated from measured IR intensity using equation (2).

It should be noted that the interfacial thermal conductance \( G(T) \) gradually increases by more than an order of magnitude with temperature in the melting range, see figure 12. The fastest growth occurs at temperatures above the solidus temperature \( T_{sof} = 805 \text{ K} \). The ITC remains stable at temperatures above the liquidus temperature \( T_{led} = 901 \text{ K} \), when the alloy is completely liquid. Interestingly, such a gradual change in \( G(T) \) is not characteristic of pure metals. The melting of a pure metal usually begins on its surface, when a thin layer of liquid is forced on the surface just below the melting point [34]. Usually, a sharp jump in \( G(T) \) occurs just before the melting point of the pure metal, when the surface of the sample becomes liquid, as was observed in experiments with pure tin, aluminum, copper, and stainless steel [20, 21]. However, a gradual increase in ITC with temperature was observed for pure metallic indium in a certain range of premelting temperatures [22]. The gradual change in \( G(T) \), found for indium and AA7075 microdroplets, indicates a gradual change in the strength of the interaction between the sample and the substrate during premelting and melting the sample.
interaction strength depends on the short-range structure of the melting sample, which can change with temperature and lead to temperature changes in the thermal contact conductance [35].

5. Conclusions

Ultrafast nanocalorimetry combined with high-speed IR thermography makes it possible to measure ITC during fast phase transformations in metal microdroplets with sub-millisecond time resolution. This method can be used to measure nanogram microdroplets in fast thermal processes at a temperature scan rate in the range of $10^3\ \text{K s}^{-1} \leq R \leq 10^5\ \text{K s}^{-1}$. The temperature difference at the interface between the sample and the solid substrate can be measured and used to determine the ITC. This temperature difference can reach 80 K during the solidification of microdroplets in an experiment with laser heating. This finding is very important for the modeling and optimization of laser-assisted technological processes.

The interfacial thermal conductance $G(T)$, being an important parameter for various industrial processes, can vary over a wide range depending on the temperature and state of the sample. The interfacial thermal conductance $G(T)$ measured for AA7075 microdroplets gradually increases by an order of magnitude with increasing temperature in the range from the solidus temperature $T_{\text{SS}} = 805\ \text{K}$ to the liquidus temperature $T_{\text{LIQ}} = 901\ \text{K}$ of the alloy. The fastest growth of $G(T)$ occurs in the melting range near the liquidus temperature. After that, $G(T)$ remains stable at temperatures above the liquidus temperature $T_{\text{LIQ}} = 901\ \text{K}$, when the alloy is completely liquid. The gradual change in $G(T)$ found for AA7075 microdroplets indicates a gradual increase in the adhesion of the sample to the substrate during melting. This adhesion depends on the short-range structure of the melting alloy; this short-range structure can change with temperature and lead to temperature changes in the thermal contact conductance [35]. The observed gradual change in $G(T)$ can be of interest for various technological processes. This result can be used in the design of materials produced in laser additive manufacturing.

The unusual melting behavior of ITC found for AA7075 microdroplets is important for understanding laser-assisted technological processes. We assume that a gradual change in ITC during melting may be characteristic of alloys with a wide melting range from solidus to liquidus temperature. Thus, it is interesting to carry out similar measurements for different alloys with a wide melting range. An interesting side observation of this work—the aluminium alloy particle remains spherical even after being heated up to 100 K above its melting point. That means the interface between particles may hold to quite high temperatures and shall be considered when modeling the additive manufacturing process. E.g., in combinations of DEM (discrete elements method) and CFD (computational fluid dynamics), typically used for that [36]. A further dependence of ITC on the surface modification of both the powder particle and the substrate shall be investigated.

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The heat flow from the membrane surface to the gas is equal to the product of the heat loss parameter $\alpha_g$, surface area $S$, and the temperature difference $(T_m - T_l)$, where $\alpha_g$ is proportional to the thermal conductivity of the gas $G_l$. In the calorimetric sensor, the distance from the membrane to the thermostat is large compared to the radius of the membrane heater $r_l = 75 \mu m$. Because of the nearly spherical temperature distribution in the gas around a small central hot zone of the membrane, this zone can be considered as a point source of the heat flow into the gas. In this case, heat flux transferred from the unit area of the central zone of the membrane is equal to $\lambda_g (T_m - T_l) / r_l$. This result was confirmed experimentally with calorimetric sensors of various sizes $r_l$ with an error of less than 30% [18, 20, 22, 37–39]. Thus, the heat loss parameter $\alpha_g$, due to the thermal conductivity of the surrounding gas, is equal to $\lambda_g / r_l = 813 \text{ W m}^{-2}\text{K}^{-1}$ at $r_l = 75 \mu m$ and $\lambda_g = 0.061 \text{ W mK}^{-1}$ of nitrogen gas at 900 K [40].

The convective component $\alpha_c$ of the heat transfer through the gas is significantly less than $\alpha_g$ at micrometer sizes. the ratio $\alpha_c / \alpha_g = N u$ decreases with the radius $r_l$ as $r_l^{-7/4}$, where the Nusselt number $N u$ is equal to $(Gr)^{1/4}/(Pr)$, and the function $f(Pr)$ with the Prandtl number $Pr$ in the case of gases is approximately equal to 1 [41]. The Grashof number is $Gr = (\rho_g \Delta T g r m^4 (r + n_t)/r)$, where $g$ is the gravitational acceleration, $\nu_g$ is the kinematic viscosity, and $\rho_g$ is the density of the gas [41]. Thus, we obtain $Gr = 2.5 \times 10^{-4}$ for nitrogen gas at $T = 900 \text{ K}$ and $\nu_g = 1.0 \times 10^{-4} \text{ m}^2\text{s}^{-1}$ [40]. Therefore, $(Gr)^{1/4} = 0.126$ and the convective contribution to heat loss from the upper surface of the membrane is about 12.6% of the heat loss parameter $\alpha_g = 813 \text{ W m}^{-2}\text{K}^{-1}$ due to the thermal conductivity of nitrogen gas at 900 K.

The contribution of thermal radiation to the heat loss parameter is $\alpha_r = \varepsilon \sigma (T^4 - T_l^4)/(T - T_l)$, where the Stefan–Boltzmann constant $\sigma = 5.67 \times 10^{-8} \text{ W m}^{-2}\text{K}^{-4}$ [40], and the emissivity of the silicon-nitride membrane $\varepsilon$ is about 0.2 [42, 43]. Therefore, $\alpha_r$ is about 1.5% of $\alpha_g$ at $T = 900 \text{ K}$ and $T_l = 300 \text{ K}$.

Thus, the parameter $\alpha_g$ of the heat loss from the upper and lower surfaces of the membrane is equal to $(1 + 0.126 + 0.015) (\lambda_g / n_t) = 928 \text{ W m}^{-2}\text{K}^{-1}$ and $1.015 (\lambda_g / n_t) = 826 \text{ W m}^{-2}\text{K}^{-1}$, respectively.

Let us estimate the sample temperature $T_m^{\text{max}}$ at $t = t_{\text{max}}$, when the membrane temperature is equal to $T_m^{\text{max}}$: 1050 K and the heating rate $R$ is zero. In this case, the heat flow from the membrane to the sample $q_m = G_m (T_m^{\text{max}} - T_m^{\text{max}})$ is equal to the heat loss from the sample to the gas $q_g = \alpha_g (T_m^{\text{max}} - T_l) S$. Therefore, $T_m^{\text{max}} - T_m^{\text{max}} = \alpha_g (T_m^{\text{max}} - T_l) S / G_m$, where $\alpha_g = 928 \text{ W m}^{-2}\text{K}^{-1}$, $S = 6.6 \times 10^{-10} \text{ m}^2$, and $G_m = 6 \times 10^{-3} \text{ W K}^{-1}$. Thus, we obtain $T_m^{\text{max}} = 1042 \text{ K}$ at $T_m^{\text{max}} = 1050 \text{ K}$.

In case of the laser heating of the sample, the radius of the hot zone is equal to $r_g$. Thus, the heat loss parameter $\alpha_{gl}$, due to the thermal conductivity of the surrounding gas, is equal to $\lambda_g / r_g$. The convective contribution to the heat loss from the sample surface is about 2.8% of $\alpha_{gl}$ at $r_l = 10 \mu m$ and $\lambda_g = 0.061 \text{ W mK}^{-1}$ of nitrogen gas at 900 K [40]. The contribution of thermal radiation to the heat loss $\alpha_r$ is equal to 0.2% of $\alpha_{gl}$ at 900 K. Therefore, in case of the laser heating, the parameter $\alpha_{gl}$ of the heat loss from the sample surface is equal to 1.03($\lambda_g / r_g$) = 6149 W m$^{-2}$K$^{-1}$.

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