Thermal Effects on the Crystallization Kinetics, and Interfacial Adhesion of Single-Crystal Phase-Change Gallium

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Although substrates play an important role upon crystallization of supercooled liquids, the influences of surface temperature and thermal property have remained elusive. Here, the crystallization of supercooled phase-change gallium (Ga) on substrates with different thermal conductivity is studied. The effect of interfacial temperature on the crystallization kinetics, which dictates thermo-mechanical stresses between the substrate and the crystallized Ga, is investigated. At an elevated surface temperature, close to the melting point of Ga, an extended single-crystal growth of Ga on dielectric substrates due to layering effect and annealing is realized without the application of external fields. Adhesive strength at the interfaces depends on the thermal conductivity and initial surface temperature of the substrates. This insight can be applicable to other liquid metals for industrial applications, and sheds more light on phase-change memory crystallization.

Understanding the crystallization process is essential in many research areas, such as phase-change (PC) memory,[11,12] protein crystallization,[13] biomineralization,[14] pathologies,[15] and semiconductors.[16] Crystallization occurs in two steps: nucleation and growth.[3,7] The nucleation of liquid is ubiquitous in nature and facilitated by active sites, such as impurities and surface irregularities (e.g., cleavages, atomically sharp wedges, and pores).[7–10] Nucleation plays a crucial role in PC materials, where structural changes from amorphous to crystalline state is triggered by electrical or optical stimulation. For instance, Loke et al.[2] reported an ultrafast crystallization speed of 500 ps in Ge2Sb2Te5 (GST) material by applying a constant low voltage via prestructural ordering (incubation) effects. Simpson et al.[11] demonstrated an optical excitation with laser-based time-resolved pulses to investigate the interfacial crystallization of superlattices in GeTe–Sb2Te3 in forming extended single-crystalline texture in supercooled liquids due to surface layering of atoms, molecules, and charged colloids adjacent to the solid walls of substrates.[6,19–22] In addition, single-crystal texture formation through physical confinement and uniaxial magnetic field have been demonstrated.[23] However, direct experimental evidence of the link between the interfacial temperature and the surface layering effect is still missing. Similarly, supercooled liquid gallium (Ga) exhibits surface layering in contact with a hard wall of a diamond substrate.[24] Ga can be supercooled to ~28 °C, or 58 °C below its freezing point (29.8 °C).[25] Because of its cost effectiveness and accessibility, Ga is an interesting material in phase transition (PT) studies. Ga also shows a promising potential in monatomic PC memory devices for data storage,[26] superconductivity,[27] metamaterial,[28] and reversible adhesion.[29] To reverse the supercooling of liquid Ga, external perturbations are applied to induce nucleation and subsequent crystal growth.[21,27,10] Even though molecular liquids are promising candidates to study phase transformation at ambient pressures and moderate temperatures, it is rather difficult to practically monitor the exact site of nucleation, since nucleation is a stochastic process.[13–17]

In this work, we study the crystallization of supercooled liquid Ga and vividly reveal the temporal change of its liquid-to-solid PT at the interface between the liquid Ga and different hard substrates. As depicted in Figure 1, the supercooled liquid Ga droplet is attached to a loadcell (see the Experimental Section). The device was lowered gently onto the substrate surface at a constant speed of 200 μm s⁻¹, attaining a compression load of 10 mN. The supercooled Ga droplet conformed to the substrate forming circular and radial wrinkles at the interface.[31] The wrinkling instability emerged at the interface to relieve the applied compressive stress. The crystallization of the liquid...
droplet was realized through a contact with a seeding crystal of solid Ga. We visualized the spatiotemporal evolution of the crystallization dynamics on transparent glass, indium tin oxide coated glass (ITO-glass), and zinc selenide (ZnSe) substrates (Figure 2; Movies S1, S2, and S3, Supporting Information). The crystallization speed depends on the surface-to-volume ratio of the Ga and the substrate capacity to act as a heat sink (Figure S1, Supporting Information).

Figure 2a–c shows the snapshots of the crystallization front and the corresponding time lapse of the crystallized fraction of Ga on the substrates. When compared to the glass substrate with low thermal conductivity (0.94 W m⁻¹ K⁻¹), the crystal growth rate was fast on the ITO-glass substrate owing to the high thermal conductivity of the ITO coating (10.2 W m⁻¹ K⁻¹). Similarly, a fast crystal growth rate was obtained on the ZnSe substrate (18 W m⁻¹ K⁻¹). We used an image-processing algorithm (python) to track the crystal front of a 1.75 mm diameter droplet through the transparent contact interfaces (Figure 2d). The Johnson–Mehl–Avrami–Kolmogorov (JMAK) equation was used to calculate the volume fraction crystallized, X, which increases with time t according to

\[ X = 1 - \exp[-(kt)^n] \]

where \( n \approx 3 \). Further, as the droplet mass increased, slower growth rate was observed due to the dependence of the substrate capacity to dissipate heat during the crystallization process (Figure S2, Supporting Information). To understand the physical mechanism of heat dissipation at the substrate–droplet interface, we used the heat conduction model reported by de Ruiter et al. The model predicts the droplet interfacial temperature \( T_{d,int} \) upon crystallization as

\[ T_{d,int} = T_{d,0} + \left( T_{d,0} - T_{s,0} \right) \frac{1}{1 + \epsilon_i/\epsilon_d} \]

where \( \epsilon_i = (K \rho C_p)^{1/3} \) is the effusivity (\( i = s \) for substrate and \( i = d \) for droplet), \( T_{d,0} \) and \( T_{s,0} \) are the initial droplet and surface temperature, respectively, \( K \) is the thermal conductivity, \( \rho \) is the density, and \( C_p \) is the heat capacity.

In Figure 3a, we also performed crystallization experiments on different transparent and nontransparent substrates with higher thermal conductivity including sapphire, silicon, and copper to verify the heat dissipation mechanism based on the thermal conduction model. Note that, in the case of Ga, we assumed that the initial droplet temperature was the same as the fusion temperature due to the induced supercooling effect. On the dielectric glass surface, the heat released during fusion was trapped at the interface, and therefore the interface temperature was elevated close to the melting temperature \( T_m \) of Ga. However, heat is rapidly dissipated at the interface when the thermal conductivity of the substrate is high (e.g., ZnSe, sapphire, silicon, and copper substrates). The heat released by the Ga droplet is dissipated via the substrate through thermal diffusion. The thermal conductivity of the substrate limits how much heat can be dissipated. The thickness of the substrates was chosen to satisfy the requirement for a semi-infinite surface (Tables S1 and S2, Supporting Information).

Different wetting behavior of Ga on the substrates resulted in a slight variation in contact angle measurements (Figure S3a, Supporting Information). Therefore, the apparent contact area measurement showed a negligible variation within the transparent substrates (Figure S3b, Supporting Information). The crystallization time decreased with decreasing thermal conductivity (Figure 3b). To characterize the interfacial adhesion of the supercooled Ga, we obtained force measurements of the crystallized Ga droplets. In situ temperature measurement enabled precise characterization of adhesion as shown in Figure 3c. The temperature profile was crucial in determining the starting and ending of crystallization, especially when the substrate was opaque. We harnessed the heat dissipated by the resistor attached to the device to melt the Ga droplet. The melting process was a sharp first-order transition at a fixed temperature under constant pressure. Supercooling of about 29 °C was induced after superheating the droplet by 7.7 °C. A Peltier device was used in Figure 3c to cool down the liquid Ga droplet to 0 °C without undergoing solidification. The crystallization was only achieved by seeded growth. The driving force of crystallization (the Gibbs free energy difference between the crystalline phase and the supercooled liquid, \( \Delta G \)) is related to the latent heat of melting \( \Delta H_m \) and the melting temperature \( T_m \) by the equation:

\[ \Delta G = \Delta H_m \frac{\Delta T}{T_m} \]

where \( \Delta T \) is the supercooling temperature \( (T_m - T) \) and \( \Delta H_m = 5585 \text{ J mol}^{-1} \) for Ga. The used seeding Ga crystal had surface irregularities and sharp wedges of the lamella morphology (Figure S4, Supporting Information). We retracted the loadcell at a constant speed of 200 μm s⁻¹ once the crystallization was complete in order to measure the adhesion between the solid droplet and the substrate (Figure S5 and Movie S4, Supporting Information). The robustness and repeatability of the seeding method was investigated in Figure S6 in the Supporting Information. Energy-dispersive X-ray (EDX) spectroscopy of both the seeding crystal and the crystallized droplet were used to evaluate the composition of Ga (Figure S7, Supporting Information). The adhesion characterization of Ga in both liquid and solid states measured on different substrates is shown in Figure 3d. The adhesion reduced drastically on the thermally conducting substrates such as silicon and copper due to fast crystallization kinetics and...
Figure 2. Contact interface and crystallization front visualization and characterization. a–c) Inverted optical microscopy video snapshots of the interface through optically transparent substrates showed seeded crystallization kinetics at the interface. The depicted images are phase-contrast optical microscopy images of the crystallization. The front propagated from right side (point of contact) to the left side of the images. The snapshots of crystallization kinetics were taken from individual droplets using a 1.75 mm post diameter at the interface of glass (a), ITO-glass (b), and ZnSe (c) substrates. b–i) The yellow dashed line represents the outline of the seeding crystal seen in the right side of the contact interface of ITO-glass. A similar seeding method was used for glass and ZnSe. Time lapses of the crystallization kinetics appeared on each corresponding image. Scale bars are 400 µm. d) Fraction of the crystallized Ga on glass, ITO-glass, and ZnSe substrates.
peeling of the crystallized droplet. Thermal stress is dominant over adhesion stress at the interface of silicon and copper. Moreover, by keeping the surface temperature high, close to Ga melting point, the peeling effect during crystallization was reduced and the adhesion increased even on copper substrate (Figure S8, Supporting Information). To grasp better understanding of the adhesion results, surface roughness effect was evaluated by measuring adhesion on a rough frosted glass. The adhesion reduced by an order of magnitude on the rough glass compared to the smooth one due to high-amplitude roughness (root-mean-square roughness of around 1.41 µm) (Figure S9, Supporting Information), which reduced the real contact area of the liquid Ga on the surface. Despite low adhesion of Ga on rough surfaces, it is much better than dry elastomeric adhesives on rough surfaces due to better conformity.\[35\]

Interestingly, when quenched at a high temperature, the crystallized Ga on ZnSe and sapphire conformed to the substrates with an extended smooth crystal structure due to a slow growth rate (Movies S5 and S6, Supporting Information). On the contrary, the crystal growth was multifaceted (dendritic) when quenched at a low surface temperature, which resulted in a fast growth rate (Movies S7 and S8, Supporting Information). Two key factors that gave rise to the smooth and dendritic crystal formation in the supercooled liquid Ga are worth elaborating. First, surface layering of the adjacent atoms or molecules in the supercooled liquid provided the pathway for the formation of the extended single-crystal. Since surface layering is not however, restricted only to the hard wall of the substrate, it could occur at the interface of the nanometer-thick oxide skin of the liquid metal, which can serve as a precursor for the single-crystalline formation at the interface.\[20,36\] Second, slow growth rate as a result of limited heat dissipation contributed to the formation of the smooth single-crystalline texture through instantaneous annealing effect of the low melting point Ga at the
interface. This typical crystallization scenario is prevalent on the dielectric glass substrate, where heat released during fusion was trapped continuously at the contact interface. As mentioned previously, the implication of instantaneous annealing on ZnSe and sapphire was verified in Movies S5 and S6 in the Supporting Information. On these surfaces, conventionally, the fast cooling rate is due to their capacity to dissipate thermal heat because of their high thermal conductivity. By keeping the surface temperature at 27 °C (0.9 \( T_m \) of Ga) throughout the crystallization process, the crystal growth was similar to the smooth single-crystalline Ga seen on the dielectric glass substrate, with crystallization front propagating across the liquid in one direction forming a uniform crystal layering on the substrates.

In addition, we verified the crystalline structure of an extended solidified Ga on large-area substrates. The supercooled liquid was confined between the substrate and a glass-capping layer (Figure S10, Supporting Information). The crystal was grown via the seeded growth method from the liquid. X-ray diffraction (XRD) measurements revealed a highly ordered, extended single-crystalline structure of \( \alpha \)-Ga when crystallized between glass/glass or sapphire/glass-capping (Figure 4a). The 2\( \theta \) scan using CuK\( \alpha \) radiation showed strong peaks of (020), (040), and (060) preferred orientation from the (010) family. The room temperature crystallized bulk \( \alpha \)-Ga had an orthorhombic crystal structure with space group \( \text{Cmca} \) (\( a = 4.519 \) Å, \( b = 7.657 \) Å, \( c = 4.526 \) Å) and contained eight atoms in the conventional unit cell. The calculated d-spacings were \( b/2 = 3.8 \) Å, \( b/4 = 1.9 \) Å, and \( b/6 = 1.3 \) Å, which agree with the reported literature values. The preference of the single-indexed crystal planes of (010) family on the substrate could be attributed to the surface energy minimization. The formation energies with respect to \( \alpha \)-Ga of (010) orientation (0.36 eV per atom) was lower than that of (100) orientation (0.64 eV per atom). This indicates better stability of (010) at room temperature up to \( T_m \). The atoms between two Ga layers along (010) are bonded covalently and show directionality with a mixture of covalent and metallic properties. In a previous study, it was reported that a perfect single-crystal structure of solid Ga could form on sapphire substrate in a nanodroplet of supercooled Ga. In addition, the XRD measurements revealed a repeating ordered structure with a peak at 38.2° and 44.4° when crystallized on ITO glass, ZnSe, silicon, and copper. Figure 4b shows the enlarged (010) peak (cyan rectangle) from the sapphire substrate. To understand the thermal behavior of the crystallized Ga, we performed differential scanning calorimetry (DSC) analysis for the crystallized Ga on different substrates. The analysis from DSC analysis results show the existence of a glassy structure with a glass transition (\( T_g \)) at around 15 °C (Figure 4c). Also, the cooling curve of the DSC measurement shows the possible oxide formation during isothermal crystallization process due to low melting point of Ga (Figure S11, Supporting Information). Detailed study on the influence of

![Figure 4](image-url)
the oxide formation during crystallization is a future work. Scanning electron microscopy (SEM) image of a fast-quenched droplet on silicon substrate shows a localized nanoscale spatial ordering in the fractured cross-section of the glassy structure (Figure S12, Supporting Information).

The PC property of Ga could be a potential tool in designing a caloric capacitor that can store unused thermal energy to perform a useful work in dynamic electromechanical devices. The interface characteristics revealed will encourage better evaluation of energy efficient PC materials with a minimum phonon scattering and less power dissipation, and paves the way for surface induced single-crystal formation from supercooled liquids. This finding could validate the temperature dependence of surface layering in supercooled liquids.

Experimental Section

Printing of the Post: A cylindrical polymer (VeroClear material) post was printed using a 3D printer (Objet260 Connex, Stratasys Ltd.). The post has outer diameter of 3 mm, inner diameter of 2.5 mm, and a height of 5 mm. A hole cavity placed at the center along the length of the post compartmentalized an electrical resistor for heating purpose (see Figure 1a). The printed post was thoroughly cleaned in 1 M NaOH solution. It was then attached to a threaded metal holder to facilitate attachment onto a force sensor for adhesion measurement. A silicone adhesive (Sil-Poxy, Smooth-On Inc.) was applied between the metal and the base part of the plastic post. After curing at room temperature for 30 min, the two parts were strongly bonded. As previously reported in,[31] 1.75 mm diameter post was replicated using double molding approach to fabricate a holder for a hemispherical droplet cap of 1.75 mm used in the crystallization study in Figure 1b.

General Handling Warning: Ga-based liquid metals corrode metal surfaces, especially aluminum (Al). Therefore, careful handling of Ga and its alloys is recommended to avoid damage to instruments and equipment including microscopes and all devices composed of Al components.

Deposition of the Liquid Ga Droplet onto the Post: A pool of melted Ga was prepared by heating commercially available pure liquid Ga (Gallium, 99.99%, ACROS Organics) to 65 °C. After heating to this temperature, supercooling effect was induced. To verify the supercooling, the molten droplet was cooled down to 0 °C without undergoing crystallization from room temperature. 44 mg of the supercooled Ga was injected into the cavity in the center of the post, which then bulged at the tip of the post forming a hemispherical cap. The injected liquid metal was in thermal contact with a resistor inserted halfway in the cavity. This resistor is used to dissipate heat via Joule effect, to melt the Ga droplet. Finally, the post was mounted onto the force sensor with the liquid metal droplet attached.

Adhesion Measurement Setup: A customized adhesion measurement setup mounted on an inverted optical microscope (Axio Observer A1, Zeiss) with a video camera (Grasshopper3, Point Gray Research Inc.) was used to visualize the phase contrast images at the contact interface. The force between the sample and the substrate was measured by a high-resolution load cell (GSO-25, Transducer Techniques). The load cell was mounted to a high-precision piezo motion stage (LPS-65 2″, Physik Instrumente GmbH & Co. KG) in the vertical direction, with a resolution of 5 nm and maximum velocity of 10 mm s⁻¹. A long-ranged motor stage (M-605 2DD, Physik Instrumente GmbH & Co. KG) was used in y direction with 1 μm resolution and high maximum velocity up to 50 mm s⁻¹.[31]

Humidity and Temperature Control: Both environmental temperature and humidity were monitored using a commercially purchased sensor (Humidity/Temperature/Dew Point Meter, Fisher Scientific). The humidity ranged from 26% to 45% and the room temperature was 22~25 °C. A six-channel handheld temperature data logger (OMEGA RXDL6DS, Omega Engineering, Inc.) was used to monitor in situ temperature of both Ga and the surface. Heating and cooling temperature controller (BTC-1-100 and BTC-SLM, Bioscience Tools) was used to regulate the environment temperature. To harvest dissipated energy for direct heating of Ga, a 220 Ohm resistor was connected in series to a 9 V power source. The heat energy dissipated by the resistor can raise up the temperature of the droplet instantaneously up to 60 °C, which is enough to melt the solid Ga instantly (the Ga melting temperature: 29.8 °C).

Microscopic Characterization: A scanning electron microscope (Zeiss Ultra 500 Gemini SEM, Carl Zeiss Inc., Oberkochen, Germany) was used to characterize the seeding crystal surface and the area of the Ga droplet, which contacted the substrate. Energy-dispersive X-ray spectroscopy (Bruker, Billerica, MA) was performed using an accelerating voltage of 15 keV on the seeding crystal and the solidified Ga droplet. 3D laser scanning microscope (Keyence VK-X200) was used for characterizing surface topology of the substrate.

XRD Measurements: XRD characterization was obtained with D8 Advance Bruker using a Cu-Kα (λ = 1.5418 Å). A glass substrate and the crystallized Ga were cooled in liquid nitrogen prior to XRD measurement to prevent any structural disorder such as surface melting of the solid crystal. Lattice constants for XRD measurements were determined from ICDD card no: 03-065-2493 for Ga. All samples were prepared at ambient room temperature.

DSC Analysis: Thermal analysis were obtained by Discovery DSC 2500 (TA Instrument). Heating or cooling rate of 5 °C min⁻¹ was used to detect the endothermic and exothermic peak for the samples that were crystallized on different substrates. The crystallized sample mass was around 5 mg.

Substrate Materials: All the substrates utilized in the experiments are commercially available. Zinc selenide, sapphire, and silicon substrates (discs) with 5 mm thickness and 25 mm diameter (Thorlabs, Inc.), a glass slide of 1 mm thickness and 120~160 nm thick ITO-coated glass slide (Sigma Aldrich), and a copper plate ≥ 99.9% (metal basis) with a thickness of 6.35 mm (Alfa Aesar) were used as the substrate materials. The copper plate was polished to maintain the same magnitude of nanoscale roughness across all target surfaces. Root-mean-square (RMS) roughness values of the smooth glass, ITO coated glass, ZnSe, sapphire, silicon, and polished copper substrates are 33, 61, 43, 39, and 105 nm, respectively. The RMS roughness of frosted glass substrate is 1.41 μm.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

M.Y. and M.S. proposed the research. M.Y., A.L., and M.S. designed the experiments. M.Y. performed the single-crystal experiments. M.Y. and A.L. performed the adhesion measurements. M.Y. and A.L. analyzed...
and interpreted the data with feedback from M.S. M.S. supervised the research. M.Y. wrote the manuscript with input from A.L. and M.S. All authors revised and commented on the manuscript.

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