Variable contact surface gripping technique for microsized objects

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A special approach is presented regarding an ice-gripping technique for microsized objects, from the initial idea to the test result. This approach uses water vapour under its triple point for ice forming when gripping or releasing the object. These conditions eliminate liquid water and cause deposition and sublimation to occur. The cooling of sharp apex tips is made possible in this way and eliminates great surface tension of liquid water that makes releasing difficult. The experimental setup and its components are described. It has been shown that this principle is capable of gripping, moving and releasing objects of 40 μm in size, requires no external intervention (such as electron beam-induced deposition) for releasing the object, is very simple and cost-effective and is shape and orientation insensitive.

1. Introduction: Over recent years, several research groups throughout the world have tried to come up with a robust and convenient physical principle that would be suitable for manipulating micro and nano-objects. Manipulation in such a manner relates to top–down methods, where tools are normally used that are in direct contact with an object and move it in the conventional way. The main difficulty when manipulating tiny objects is not the gripping itself but the releasing of the object because of a large adhesion force (van der Waals force), which is much greater than the gravitational force for small (<100 μm) objects. Avoiding or minimising these forces requires unconventional ways of gripping [1], which are provided by optical tweezers [2, 3], two-finger grippers [4], atomic force microscopy [5, 6] and scanning tunnelling microscopy [7, 8]-based techniques, ice-grippers [9–11] etc. The adhesion force does not take effect when manipulating with optical tweezers or magnetic tweezers but they do have certain other limitations, such as lack of selectivity and exclusivity (optical tweezers) or need of a magnetic object [12]. When applying other techniques such as a two-finger gripper, adhesion-gripper or any other, where the gripper comes into direct contact with the object, a special approach has to be used which overcomes the adhesion issue, such as [13–16] etc. Special coatings for minimising these effects do not eliminate the adhesion problem, only minimise it. The plunger, acceleration and vibration approaches are also inappropriate, since the objects can fly off in an uncontrollable way. Some authors have also reported releasing of an object using electron beam-induced deposition (EBID), where the object is soldered to the surface where it should be released [17]. Walle *et al.* [9], Ru *et al.* [10] and Liu *et al.* [11] have developed grippers using ice that encloses the object for manipulation. Walle *et al.* [9] developed a system that operated within a liquid (water), where they cooled the tip with a Peltier element. Ice formed around the tip surrounding the object of interest, and it could then be moved freely. When it was necessary to release it, the tip was simply heated-up and the ice melted. It was tested on an object 600 × 600 μm in size. The problem here could be cooling of the small tips because of the relatively high thermal conductivity and heat-capacity of the liquid, which might not allow really small tips (<1 μm) to be cooled. Ru *et al.* [10] and Liu *et al.* [11] have reported the development of an ice-gripper that operates within air and cools down the tip with a small amount of liquid water on the tip's surface, when in contact with an object. This system has only been tested on relatively large objects (300 μm) [10] or a couple of millimetres [11].

This Letter presents an experimental technique aimed at gripping micro or even nanosized objects, where ice deposition and sublimation are used when gripping or releasing the object. In this approach the tip is cooled down near the object, ice then deposits around the tip and encloses the object when gripping. When releasing the object, the tip is heated-up and the ice sublimates. A vacuum environment allows for cooling-down of very sharp tips (apex diameter under 1 μm), thus making possible reliable gripping and releasing of objects within a size range of only a few micrometres. The presented system is shape and orientation insensitive, cost-effective, reliable and simple.

2. Gripping/releasing procedure: For an ice-gripper system that uses water vapour as a building block for ice-formation, the water vapour would have to be within a vacuum of under 6 mbar (triple point of water). Under such conditions, the stable phases only contain solid ice and water vapour (Fig. 1). The procedure starts with gripping, when the tip is converged towards the object and cooled down so that ice is formed (deposited) around the object and the tip. When released, the tip is heated-up and the ice sublimates to the environment's physical state. The following presented procedure has proved to be useful for gripping, moving and releasing objects:

1. Move the tip close to the object.
2. Lower the table and tip temperatures to just above the deposition point (for example 0.2 mbar and −30°C).
3. Cool-down the tip (for example −50°C), wait until the ice surrounds the object.
4. Touching the object with the ice (the object is gripped).
5. Move the object to the desired position on the table.
6. Heat the tip and table and wait until all the ice/water sublimates.

It has been shown that this gripper works as a variable contact surface tip and, consequently, as a variable adhesion force tip, since the adhesion force is proportional to the contact surface between two objects. This can be met by the phenomena of the van der Waals forces [18], which are normally attractive forces. van der Waals force (F_W) between two objects comes from the derivative of the free-energy between two objects

$$F_W = -\frac{dG}{dl} \tag{1}$$

where l is the distance between the objects. The interactive free-energy for two parallel surfaces (contact area between ice and

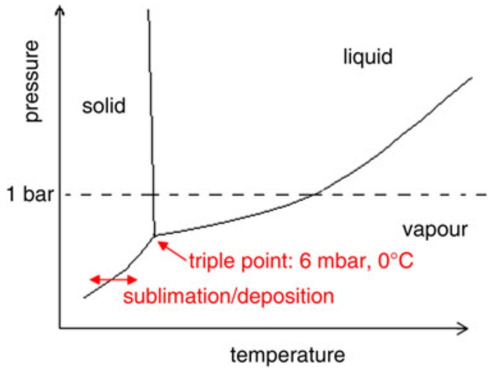

Figure 1 *Water-phase diagram*

object L^2) (Fig. 2) is

$$G_{IO} = -\frac{A_{HIO}}{12\pi l^2} L^2 \tag{2}$$

where A_{HIO} is the Hamaker coefficient between the ice and the object's material l across the vacuum, and l the average distance across the object's roughness between the ice and the object. Interaction free-energy G_{ST} between the sphere with radius R and the plane (contact case between table and object) with average distance l, and the Hamaker coefficient A_{HOT} between the object's material and the table's material across the vacuum is:

$$G_{ST} = -\frac{A_{HOT}R}{6l} \tag{3}$$

When releasing the object the tip apex is in contact with the object and the best approximation is the sphere–sphere contact case. Interaction free-energy between two spheres with radii R_1 and R_2, the Hamaker coefficient between the tip's material and object's material across the vacuum A_{TO} and the average distance across the object and tip surface roughness between the tip and the object l is

$$G_{TO} = -\frac{A_{TO}R_1R_2}{6(R_1 + R_2)l} \tag{4}$$

The condition for gripping is therefore $F_{WIO} > F_{WST}$ and $F_{WST} > F_{WTO}$ for releasing the object. Since interaction free-energy between the ice and the sphere is quantified on the unit area (2), we can conclude that the size of the F_{WIO} is proportional to the contact area between the two objects. In the case of the presented ice-gripper, the contact area between the tip and the object varies using ice when varying its size regarding deposition and sublimation (Fig. 2). On the other hand, the F_{WST} between the object and the table remains constant, so it is possible to switch between the ratios needed for gripping or releasing. It also shows that the van der Waals force is the more likely dominant force that makes gripping possible and not the mechanical shape coupling between the ice and the object. This is due to the fact that it was practically impossible to grip the object before the ice surface around the object was greater than that between the object and the table.

3. Description of the setup: A special system was built for the purpose of testing the ice-gripping principle operating within a vacuum. It consists of a vacuum-chamber with a vacuum-pump, the Peltier element primary-cooling of antifreeze liquid, a microscope, a vacuum-gauge with a vacuum-level controller (VLC), two water pumps for cooling, two Peltier element controllers, a small Peltier element with a copper tip attached to it, a polished table and a three degrees-of-freedom (DOF) nanopositioning stage (Fig. 3).

The vacuum system consists of a vacuum-pump, a vacuum-chamber and an evaporator. The vacuum pump used is a simple dual-stage rotary-vane vacuum-pump which continuously pumps the gases out of the vacuum vessel. At the same time water vapour is inserted by the evaporator, so the desired water vapour pressure is present. This system could reach down to 10 Pa of absolute-pressure.

The microscope consists of a ×10-long working distance objective with a ×7 zoom module that provides ×70 of optical magnification. A low-magnification objective has to be used to ensure that the image is not as distorted by the viewport's glass as strongly as it might be by a high-magnification objective. The microscope produces an estimated resolution of 1 μm. A FireWire camera is used for image acquisition.

Another important element is the vacuum gauge with a VLC. A simple thermocouple gauge (Varian type 0531) is used which has a large measurement error at higher pressures (up to 50%) but this error becomes low enough (under 10%) at pressures that are relevant for the objective (well below 1 mbar). A further problem when measuring calibration is that the gases in the vacuum vessel do not only include the air for which the gauge has been calibrated. In this case the prevailing medium water vapour and therefore an additional measuring error have to be considered. Despite this fact, this was not considered to be critical, since the goal of this research was to experimentally prove the feasibility of the new method and, therefore to present an order of useful parameters.

Primary cooling consists of Peltier elements with brass cooling bodies on each side. The hot side was cooled down with water and ice. On the cold side was a closed tube system filled with an antifreeze liquid, which runs to the cooling body of a small Peltier element in a vacuum chamber and then back to the primary cooling body. This setup can reduce the temperature of the antifreeze liquid down to approximately −15°C.

Tip-cooling and heating represented a two-stage Peltier element with ΔT = 93°C (Fig. 4). The warm-side of the element was cooled-down using the antifreeze liquid from primary cooling.

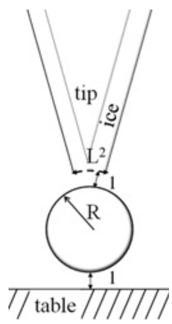

Figure 2 *van der Waals force calculation quantities*

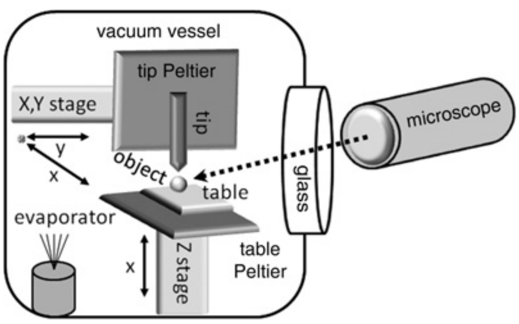

Figure 3 *Vacuum-chamber with the microscope*

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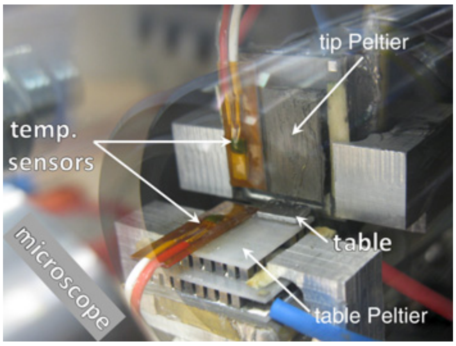

Figure 4 *Tip-cooling and table-cooling Peltier elements with temperature sensors*

Temperature reductions to −60°C were achievable using this current setup. Table cooling was the same tip-cooling Peltier element with a table attached instead of the tip. All the Peltier elements were powered through Peltier element controllers, which were connected with a PC via RS232. The tip itself was made from a copper wire 50 μm in diameter using the etching technique [19, 20], which allowed the diameter of the tips' apexes to be under 1 μm.

The last component is the three DOF nanopositioning stage. It was a set of Piezo LEGS® motors. The smallest step of 3.9 nm was achievable with current driving electronics. A linear magnetic positional encoder was installed on each motor, therefore 61 nm closed-loop positioning was possible [21, 22].

The block diagram which presents the summary of the information/power and mass flows of the setup is presented in Fig. 5.

4. Experiments and discussion: The first gripping experiments showed that it was very hard or even impossible to grip the object if the table was not properly cooled down. This occurred because of cooling of the tip to 10°C below freezing point, while the object was still placed on a room-temperature table (about 20°C). When the tip surrounded with the ice was placed in contact with the object, then the ice partly sublimated because of high temperature of the object (about 20°C). This effect made gripping impossible.

Further unexpected behaviour was that the water vapour did not freeze at the expected temperature derived from the water-phase diagram (Fig. 1), but froze at a lower temperature. Deposition and sublimation on the tip do not occur immediately, but the liquid phase (a small droplet of approximate diameter of <1 μm) also appeared shortly (<5 s) during cooling and heating. The reason was the lack of impurities within the vacuum-chamber that acted as an ice-forming nucleus. Under approximately −42°C

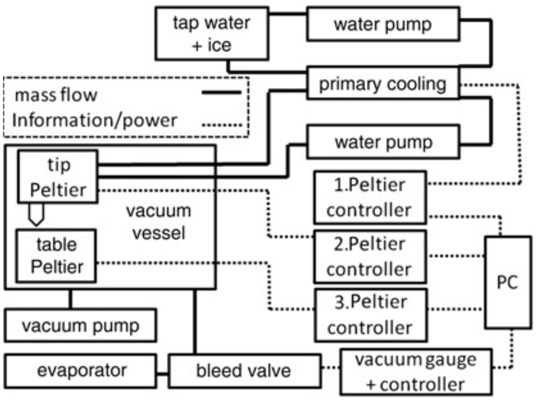

Figure 5 *Ice gripper system block diagram*

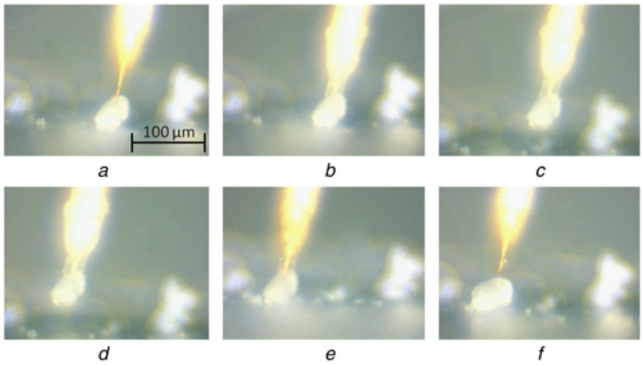

Figure 6 *Snapshots of ice-gripping flour particles*
a Closing the tip
b Ice-forming around object deposition
c Lifting the object
d Moving the object
e Releasing object-sublimation
f Tip lifting
Object size is ∼40 μm

the liquid froze, even though there was no ice nuclei present [23]. The freezing point also dropped with reducing vapour pressure (Fig. 1). The explanation was that the water-phase diagram only showed the stable phases (solid, vapour) of water under certain conditions, but not the metastable phases, which were liquid in this particular case. In the case of deposition very small droplets of liquid started to form on the tip after a short time, but once they had frozen (under about −42°C), only pure deposition occurred. Once deposition/condensation had started, the pressure began to drop (acted like a cryopump), and the opposite when heated. Therefore initial pressures in the vacuum vessel (just before the gripping procedure begins) need to be always given in this work. The VLC tried to compensate for this, but was much slower than needed. In the case of sublimation, the ice melted into a liquid, which then rapidly evaporated before the liquid water formed the droplets, which could be fatal for accurate positioning of a small-sized micro or even nanosized object. Sublimation occurred much faster than deposition. A typical time for deposition was about 5 s and for sublimation around 3 s. These durations were influenced by the pressure and tip temperature and could be used as guidance only. The gripping sequence is presented in Fig. 6. The wire used for the tip in this case had a diameter of 50 μm and a tip–apex diameter below the resolution of this microscope (<1 μm). The objects manipulated in this case were around 40 μm in size. It could be seen that with such methods, gripping, moving and releasing were possible without the intervention of any other technique (such as EBID). It has been shown that this gripping principle also works for irregular-shaped objects and not only just for spheres.

The pressures and temperatures that proved useful for gripping were much lower than initially expected. This was mostly because of the longer liquid-phase duration at higher pressures (and consequently higher temperatures). Those vapour pressures that proved to be useful were under 1 mbar and the tip temperatures under about −15°C. This configuration of parameters still offered a very high deposition rate and was therefore considered as the upper limit, so typical conditions were around 0.2 mbar and −50°C. Pressure fluctuations were kept to a minimum, since deposition conditions influenced by pressure and ice growth could be started spontaneously at constant temperature under increased pressure (the sublimation/deposition curve could be crossed) (Fig. 1). Similar problems could occur with an ice growth-rate during the gripping procedure, which could be dramatically increased and could cause a large amount of ice to be deposited on the tip.

5. Conclusion: This Letter shows that it is possible to grip, move and release microobjects using variable van der Waals forces. This technique moves the heat conductive-tip close to the object, which lies on the cooled table. After this, the tip and the table are cooled-down to the desired values, so that the ice from the tip surrounds the object. Ice-forming is enhanced because of deposition within the vacuum, where water vapour is the dominant component of the remaining gases. Then, the object can be picked-up and moved. On the other hand, releasing of the object is made by (partial) sublimation, where most of the ice sublimates into gas directly. The object is then released. It was found that the ice-gripper acts like a variable contact surface tip and, therefore as a variable van der Waals force tip. Mechanical coupling has a minor influence during the gripping phase. This Letter describes and reports the experimental testing of a very promising technique and shows successful gripping, moving and releasing of an irregular-shaped flour particle about 40 μm in size, which is a significant improvement compared with other ice grippers. The presented gripper also offers a very cost-effective solution, since it is compatible with an environmental scanning electron microscope, where only the positioning stage, a cooled tip and a cooled table have to be implemented within its chamber, which would also allow the lower object size limit to be determined. It is also object-shape and orientation insensitive and reliable. The presented variable van der Waals force gripper could overcome the drawbacks of the other micro or nanogrippers described in the Introduction section.

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