# Journal of Visualized Experiments Scanning SQUID Study of Vortex Manipulation by Local Contact --Manuscript Draft--

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Corresponding Author:	Beena Kalisky Bar Ilan University Ramat Gan, ISRAEL	
Corresponding Author Secondary Information:		
Corresponding Author E-Mail:	beena.kalisky@gmail.com	
Corresponding Author's Institution:	Bar Ilan University	
Corresponding Author's Secondary Institution:		
First Author:	Eylon Persky	
First Author Secondary Information:		
Other Authors:	Eylon Persky	
	Anna Kremen	
	Shai Wissberg	
	Yishai Shperber	
Order of Authors Secondary Information:		
Abstract:	Local, deterministic manipulation of individual vortices in type 2 superconductors is challenging. The ability to control the position of individual vortices is necessary in order to study how vortices interact with each other, with the lattice, and with other magnetic objects. Here, we present a protocol for vortex manipulation in thin superconducting films by local contact, without applying current or magnetic field. Vortices are imaged using a scanning superconducting quantum interference device (SQUID), and vertical stress is applied to the sample by pushing the tip of a silicon chip into the sample, using a piezoelectric element. Vortices are moved by tapping the sample or sweeping it with the silicon tip. Our method allows for effective manipulation of individual vortices, without damaging the film or affecting its topography. We demonstrate how vortices were relocated to distances of up to 0.8 mm. The vortices remained stable at their new location up to five days. With this method, we can control vortices and move them to form complex configurations. A reliable This technique for vortex manipulation could also be implemented in applications such as vortex based logic devices.	
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Dear Editor,

Please find enclosed our manuscript entitled "Scanning SQUID Study of Vortex Manipulation by Local Contact" that we would like to submit to the Journal of Visualized Experiments. This paper highlights a protocol for manipulation of individual vortices in thin superconducting films, using local mechanical contact.

We consider these data suitable for the Journal of Visualized Experiments, as our protocol allows effective, deterministic manipulation of vortices. Our protocol describes a new method for vortex manipulation. Our method does not require applying current, magnetic field or additional fabrication steps, and enables manipulation of vortices at a temperature range wider than previously used methods.

The techniques presented in this paper and demonstrated in video format will be highly useful for researchers working in the field of condensed matter physics, but could also be of interest to a broader scientific community, because tunable and localized control of nano-magnetic objects is essential for various applications, such as logic elements and spintronic devices. In our studies we use scanning SQUID microscopy, but this method can be easily implemented by other scanning probe techniques, such as scanning Hall probe, or scanning magnetic tunnel junction.

Anna Kremen and Beena Kalisky designed the procedures described in the manuscript. All authors performed the experiments and analyzed the data. Finally, Eylon Persky wrote the manuscript, with contribution from all authors. During the preparation and submission of this manuscript, we have been kindly assisted by Mr. Benjamin Werth.

We have provided below contact information for four potential reviewers. Thank you for your consideration of this manuscript. We look forward to hearing from you.

Sincerely yours, Beena Kalisky



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Potential reviewers:

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Alexander Gurevich Old Dominion University. Email: gurevich@odu.edu

Vladimir Kogan Iowa State University Email: kogan@iastate.edu

Charles Reichhardt Los Alamos National Laboratory Email: reichhardt@lanl.gov

#### TITLE:

Scanning SQUID Study of Vortex Manipulation by Local Contact

#### **AUTHORS:**

Persky, Eylon

Department of Physics and Institute of Nanotechnology and Advanced Materials

**Bar-Ilan University** 

Ramat-Gan, Israel

eylon.persky@gmail.com

Kremen, Anna

Department of Physics and Institute of Nanotechnology and Advanced Materials

Bar-Ilan University

Ramat-Gan, Israel

anna.ker.biu@gmail.com

Wissberg, Shai

Department of Physics and Institute of Nanotechnology and Advanced Materials

**Bar-Ilan University** 

Ramat-Gan, Israel

shaiwis@gmail.com

Shperber, Yishai

Department of Physics and Institute of Nanotechnology and Advanced Materials

**Bar-Ilan University** 

Ramat-Gan, Israel

shperber@gmail.com

Kalisky, Beena

Department of Physics and Institute of Nanotechnology and Advanced Materials

Bar-Ilan University

Ramat-Gan, Israel

beena@biu.ac.il

#### **CORRESPONDING AUTHOR:**

Kalisky, Beena

beena@biu.ac.il

#### **KEYWORDS:**

Superconductivity, vortex matter, vortex manipulation, scanning SQUID microscopy, superconducting thin films, superconducting vortices

#### **SHORT ABSTRACT:**

We present a protocol for manipulation of individual vortices in thin superconducting films, using local mechanical contact. The method does not include applying current, magnetic field or additional fabrication steps.

#### LONG ABSTRACT:

Local, deterministic manipulation of individual vortices in type 2 superconductors is challenging. The ability to control the position of individual vortices is necessary in order to study how vortices interact with each other, with the lattice, and with other magnetic objects. Here, we present a protocol for vortex manipulation in thin superconducting films by local contact, without applying current or magnetic field. Vortices are imaged using a scanning superconducting quantum interference device (SQUID), and vertical stress is applied to the sample by pushing the tip of a silicon chip into the sample, using a piezoelectric element. Vortices are moved by tapping the sample or sweeping it with the silicon tip. Our method allows for effective manipulation of individual vortices, without damaging the film or affecting its topography. We demonstrate how vortices were relocated to distances of up to 0.8 mm. The vortices remained stable at their new location up to five days. With this method, we can control vortices and move them to form complex configurations. This technique for vortex manipulation could also be implemented in applications such as vortex based logic devices.

#### **INTRODUCTION:**

Vortices are magnetic objects at the nanoscale, formed in type 2 superconductors in the presence of external magnetic field. In a defect free sample, vortices can move freely. However, different defects in the material result in regions of reduced superconductivity which are energetically favorable for vortices. Vortices tend to decorate these regions, also known as the pinning sites. In this case, the force required to move a vortex must be greater than the pinning force. Properties of vortices, such as vortex density, interaction strength and range, can be easily determined by external field, temperature, or geometry of the sample. The ability to control these properties makes them a good model system for condensed matter behavior that can be easily tuned, as well as suitable candidates for electronic applications<sup>1,2</sup>. Control of the location of individual vortices is essential for the design of such logical elements.

Mechanical control of magnetic nanoparticles had been achieved before. Kalisky et al. recently used scanning superconducting quantum interference device (SQUID) to study the influence of local mechanical stress on ferromagnetic patches in complex oxide interfaces<sup>3</sup>. They were able to change the orientation of the patch by scanning in contact, pressing the tip of the SQUID into the sample, applying a force of up to 1  $\mu$ N in the process. We have used a similar method in our protocol in order to move vortices.

In existing studies of vortex manipulation, motion was achieved by applying current to the sample, thus creating Lorentz force<sup>4-6</sup>. While this method is effective, it is not local, and in order to control a single vortex, additional fabrication is required. Vortices can also be manipulated by applying external magnetic field, for example with a magnetic force microscope (MFM) or with a SQUID field coil<sup>7,8</sup>. This method is effective and local, but the force applied by these tools is small, and can overcome the pinning force only at high temperatures, close to the critical temperature of the superconductor. Our protocol allows effective, local manipulation at low temperatures (4K) without additional fabrication of the sample.

We image superconducting vortices using scanning SQUID microscopy. The sensor is fabricated on a silicon chip which is polished into a corner, and glued on a flexible cantilever.

The cantilever is used for capacitive sensing of the surface. The chip is placed at an angle to the sample, so that the contact point is at the tip of the chip. We apply forces of up to 2  $\mu$ N by pushing the chip into the sample. We move the sample relative to the SQUID by piezo elements. We move the vortex by tapping the silicon tip next to a vortex, or by sweeping it, touching the vortex.

#### **PROTOCOL:**

#### 1. Access to a Scanning SQUID system

- 1.1. Use a scanning SQUID system that includes a SQUID sensor fabricated on a chip $^{9,10}$ , stick slip coarse motion stage, and a piezo-based scanner for fine motion. See Figure 1.
- 1.2. Polish the SQUID chip into a corner around the pickup loop. The material of the chip needs to be removed all the way to the pickup loop.
- 1.2.1. Gently polish the SQUID, using a 5 to 0.5 µm nonmagnetic polishing paper.

Note: After the polishing stage the pickup loop can be brought into close proximity, or contact, with the sample.

## 2. Deposition of Niobium (Nb) thin film with direct current (DC) sputtering.

- 2.1. Obtain a substrate. In this work, use a boron-doped silicon substrate with 500 nm of silicon oxide. Other substrates such as SrTiO and MgO are possible.
- 2.2. Reach a base pressure of  $10^{-7}$  Torr in the chamber. Pre-sputter the evaporation chamber at room temperature with a 99.95% Nb target, in an argon environment at a pressure of 2.4 mTorr with a deposition rate of 1.8 Å/s for 10 min. Note that the deposition process can start only when the base pressure in the chamber is less than  $10^{-7}$  Torr. If pressure is higher repeat the pre-sputtering stage.
- 2.3. Place substrate in the chamber.
- 2.4. Deposit Nb thin film by sputtering at room temperature from a 99.95% Nb target, in an argon environment at pressure of 2.4 mTorr with a deposition rate of 1.8 Å/s.

## 3. Sample-tip alignment

- 3.1. In this stage, align the sensor chip with the sample so that the tip of the chip makes contact with the sample when moving the vortices. To achieve this, use an alignment angle of at least 4°.
- 3.2. Glue a flexible cantilever on a conducting plate with a dielectric layer. Then, glue the SQUID chip on the cantilever. The capacitance between the cantilever and a static plate determines the contact with the sample and the extent of stress applied.
- 3.3. Load sample on the microscope. Glue the sample to a designated sample mount using a varnish or silver paste. Glue the mount to the Z piezo element (Figure 1a).

- 3.4. Connect the stick slip coarse motion system to a controller.
- 3.5. Set up optical imaging from two angles the front and the side of the chip. Use two telescopes placed on translation stages, directed to the front of the chip and one of its sides.
- 3.6. Using the Z stick slip coarse motion stage, move the sample to a distance of 1  $\mu$ m from the sensor, so that the sensor's reflection is visible on the sample.

NOTE: Contact between the sample and the sensor at this stage may harm the SQUID.

- 3.7. Move the sample 0.5 1 mm away from the sensor using the Z stick slip coarse motion stage to prevent damage to the SQUID.
- 3.8. Rotate the alignment screws (Figure 1a) to get equal front angles (i.e. angles the sides of the chip's tip make with its reflection, as seen in Figure 1c).
- 3.9. Move the sample to a distance of 1  $\mu$ m from the sensor. Check the angles and repeat step 3.7 and 3.8 if necessary.
- 3.10. Rotate the alignment screws to get an angle of 4 degrees between the sensor and the sample (Figure 1d). Make sure the tip of the chip is the part which makes contact with the sample.

#### 4. Measurements

4.1. Load the scanning head (Figure 1a) to a 4K cooling system.

Note: Scanning head should be connected to a cold plate, and surrounded by a vacuum can. Wire a coil around the can for applying external magnetic field (low fields of several Gauss are sufficient for this study). Cover this setup with a Mu-metal shield.

- 4.2. Cool in the presence of magnetic field, by applying current through the coil surrounding the microscope. Choose the field strength carefully to achieve the desired vortex density. Use  $1\Phi_0=20.7 G/\mu m^2$  to calculate the cooldown field. For example, for 10 vortices in a 10  $\mu m$  by 10  $\mu m$  area, apply 2.07 G.
- 4.3. For changing to a new vortex density heat sample above the superconducting transition temperature (For Nb, heat above 10 K). Apply the new field.
- 4.4. Cool sample to 4.2 K.
- 4.5. Turn magnetic field off. Turn SQUID on.
- 4.6. Move the sample close to the SQUID using the stick slip coarse motion system.
- 4.6.1. Apply increasing voltages on the Z- stick slip cube to move the sample closer to the SQUID chip.

- 4.6.2. Apply voltage between the cantilever and the plate for reading the capacitance using a capacitance bridge (0.1-1 V typically).
- 4.6.3. Sweep the voltage on the Z piezo element. Measure the capacitance between the cantilever and the plate. If a large change in the capacitance occurs, the sample is in contact with the SQUID chip.
- 4.6.4. If the sample did not make contact with the chip, repeat steps 4.6.1-4.6.3 until contact is observed.
- 4.6.5. Optional: Use course motion to adjust the spacing between the tip and the sample so that contact occurs at low voltages (0 10 V applied on the Z piezo).
- 4.6.6. Once there is contact, repeat steps 4.6.2-4.6.3 in several locations in order to determine the tilt angles of the surface and to define the plane of the sample, relative to the sensor.
- 4.7. Sweep the voltage on the X and Y piezo elements in order to move the sample relative to the sensor. Scan at a constant height above the sample, without contact between the tip and the sample, in order to map vortex distribution. Achieve a constant scan height by changing the voltage on the Z piezo according to the X and Y locations, and to the plane defined in 4.6.
- 4.8. Choose a vortex and scan around it to precisely determine the location of its center. Note that the vortex location is relative to the SQUID's pickup loop, not to the contact point.
- 4.9. Turn SQUID off.
- 4.10. Apply a voltage that is greater than the touchdown voltage to the z piezo and either tap next to the vortex center or sweep the vortex by dragging the sensor (in contact with the sample) slowly on the sample to a desired location. The vortex will move towards the tap or in the sweeping direction. Typical values to add to the applied z piezo voltage are 2-5 V.
- 4.11. Turn SQUID on.
- 4.12. Image again at a constant height without contact to locate the new location of the vortex.

#### **REPRESENTATIVE RESULTS:**

Our protocol was successfully tested on thousands of individual, well separated vortices in two samples of Nb, and nine samples of NbN. We generate new vortices on the same sample by heating the sample above Tc, and cooling it back to 4.2K in the presence of a magnetic field. We chose the external magnetic field to achieve the desired vortex density. We show here data from these experiments. These results have been described in detail by Kremen et al<sup>11</sup>.

The protocol described here allows for controllable manipulation of vortices into various configurations (Figure 2). Single vortices were moved over distances up to 1 mm (Figure 3), and remained stable at their new locations.

#### Figure 1. Scanning SQUID system.

(a) The scanning head. (b) Enlarged photo of the area circled in (a). (c) Sample-sensor front angle. The angles  $\alpha$  and  $\beta$  between the chip and its reflection from the sample should be equal on both sides. (d) The chosen alignment angle between the sensor and the sample. The angle between the chip and the reflection is twice the desired angle, which should be at least 4°.

## Figure 2. Manipulation of vortices to form the letter B.

(a) Initial configuration after cooling the sample in the presence of magnetic field. (b) A new configuration after moving the vortices, in the shape of the letter B.

### Figure 3. Several manipulations of a single vortex, dragging it over a distance of 820 μm.

Inset: (a) A single vortex. The keyhole shape is due to convolution between the magnetic signal and the sensor's point spread function. (b) A scan in contact with the SQUID turned on. The initial location of the vortex is at the left of the picture. The peak of the signal moves to the right with the vortex, until the vortex is relocated at the right end and no longer moves. (c) A sketch of a scan in contact. The tip of the sensor is the first to make contact with the sample, while the vortex location received from the scan is relative to the pickup loop, which is offset from the tip.

#### **DISCUSSION:**

Successful manipulation of vortices depends on several critical steps. It is important to align the sensor at an angle, such that the tip of the chip will be the first to make contact with the sample. Second, it is important to note that the force exerted on the sample is determined by the mechanical properties of the cantilever that the chip is mounted on. In the elastic regime, the force applied is proportional to the deflection, x, according to Hooke's law:

$$F = -kx$$

Where k is the spring constant, determined by Young's modulus of the material, and its physical dimensions, and is given by

$$k = \frac{Et^3w}{4l^3}.$$

Here, E is Young's modulus, t is the thickness of the beam, w is the width and I is the length. For a copper cantilever, E=117 GPa. Our cantilever was 0.017 mm thick, 3 mm wide and 10.7 mm long, which give k=0.35 N/m. When the voltage in the Z piezo was 1 V below touchdown, the deflection was 1.6  $\mu m$ . This gives a force of 0.56  $\mu N$ . It is important to choose the cantilever material and dimensions properly, to get the desired force.

It is also important to note that the location of the vortex as scanned by the SQUID is relative to the pickup loop, and that the contact point is displaced from the pickup loop according to the size of the chip and the polishing. This displacement is to be accounted for when choosing the location of the tap event, or the contact scan, to insure that the tip of the chip makes contact near the vortex location.

If a vortex was not displaced after scanning in contact, applying more stress by pushing the tip harder into the sample, pressing the sample for a longer period or dragging the tip more slowly across the sample may help overcome the pinning force and dislocate the vortex.

The sample did not show memory of the manipulation; we observed no change in the diamagnetism of the sample, corresponding to the superfluid density, as well as no change to the topography of the sample. New vortex configurations created after reheating and cooling in the presence of magnetic field did not show memory of previous manipulations either<sup>11</sup>.

Our method is limited by the size of the contact point. The technique has the potential for fine tuning the location of vortices, but so far we have demonstrated the abilities of the protocol for rather large, polished tips of the chip (from 100 nm up to 1  $\mu$ m). Characterization of the tip is needed in order to know the strain gradients.

In conclusion, our protocol allows for manipulation of individual vortices in superconducting thin films at low temperatures and without further fabrication of the sample. Mastering the ability to control the location of vortices may have applications in the design of flux based logic gates, as well as in the study of interactions of vortices with other vortices, the lattice, and other magnetic particles.

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#### **DISCLOSURES:**

The authors have nothing to disclose.

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cantilever

SQUID chip

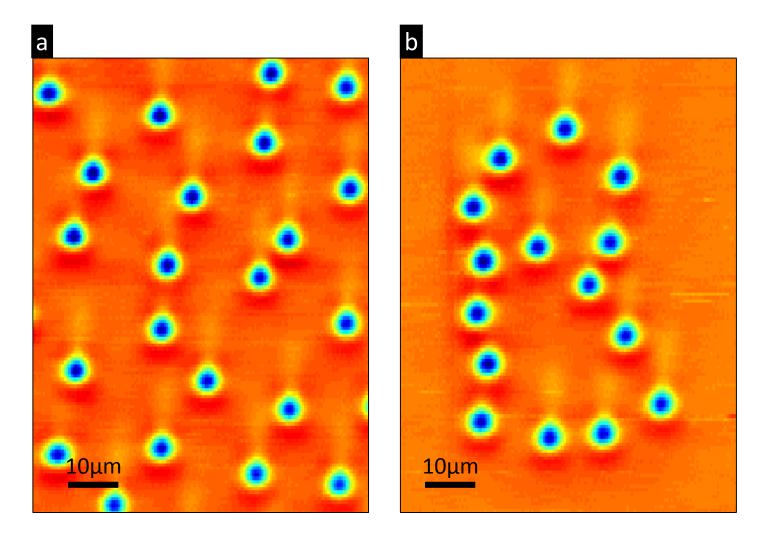
alignment screws

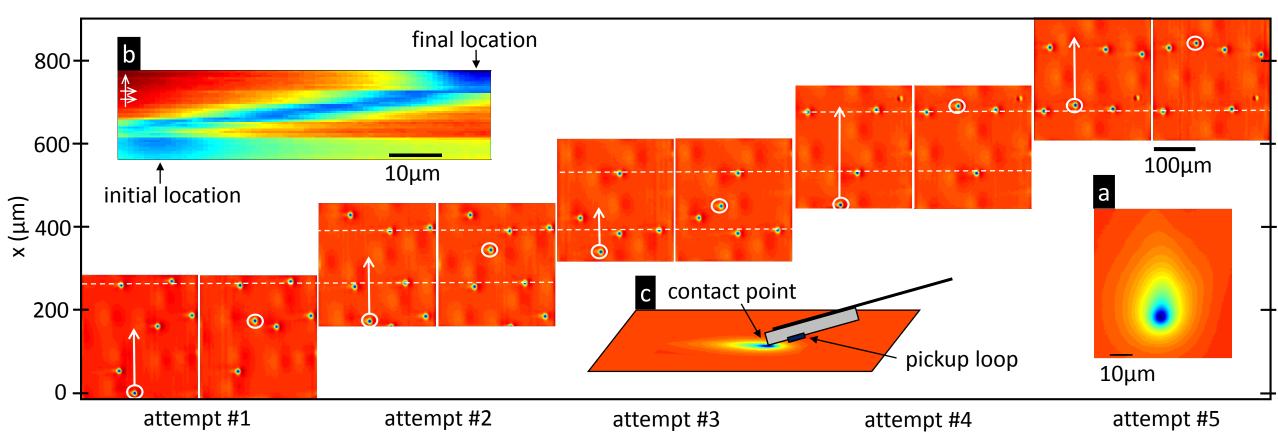
stick-slip coarse

motion system

sample

alignment screws Piezoelectric Z bender front angle Piezoelectric X,Y bender side angle cantilever SQUID chip sample





Name of Reagent/ Equipment	Company	Catalog Number	Comments/Description
stick slip coarse motion system	attocube	ANPx-101	x,y motion
stick slip coarse motion system	attocube	ANPz-101	z motion
stick slip coarse motion system controller	Attocube	ANC 300	
high voltage amplifier	Attocube	ANC 250	
data acquisition card	National Instruments	NI PCIe-6363	
piezo elements	Piezo Systems Inc	T2C	non magnetic
low noise voltage preamplifier	Stanford Research Systems	SR 560	
capacitance bridge	General Radio	1615A	
telescope	NAVITAR	1-504516	
camera	MOTICAM	MP2	
dewar	Cryofab	N/A	
insert	ICE oxford	N/A	
Mu-metal shield	Amuneal	N/A	
vacuum cap	ICE oxford	N/A	
sputtering system	AJA international Inc	N/A	
lapping film	3M	261X	non magnetic
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Silver paste	Structure Probe Inc	05063-AB	



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#### **CORRESPONDING AUTHOR:**

Name:	Beena Kalisky				
Department:	Department of Physics Institute of Nanotechnology and Advanced Materials, Bar-Ilan University, Ramat-Gan, Israe				
' Institution:					
Article Title:	Scanning SQUID Study of Vortex Manipulation by Local Contact				
Signature:	Beena Kalisky	10/5/2016 ate:			

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Dear Editor,

We thank you and the referees for the helpful comments. We have corrected the manuscript accordingly, and we submit a revised version, with the changes highlighted. In the list below, we address each comment in blue.

Changes to be made by the Author(s):

- 1. Please take this opportunity to thoroughly proofread the manuscript to ensure that there are no spelling or grammar issues. The JoVE editor will not copy-edit your manuscript and any errors in the submitted revision may be present in the published version.

  We corrected a few typos and changed some words to make sentences more clear (lines 81, 116,267,280).
- 2. Please decide between open access and standard access. On Editorial Manager, you have selected access but on the Author License Agreement, you have selected open access. We chose the green route and have been in touch with Alex Bhatty regarding some details.
- 3. Please provide a scale bar in Figure 2. We added a scale bar and resubmitted the figure.
- 4. Formatting:
- -Please define all abbreviations at first occurrence (e.g. DC).

We defined the abbreviations (lines 78,115).

-2.3 – It is unclear why Kurt J. Lesker is cited here. If the target was obtained from him, this should be indicated in the materials table. Is anything done to the sample after deposition (like any cleaning)?

We removed Lesker from the protocol (line 128) and added the target to the materials table. The sample is ready for use after the deposition.

#### 5. Grammar:

-1.2 – Please clarify what is meant by "Polish the SQUID chip into a corner located near the pickup loop". This reads as though the chip is moved into a corner by polishing it. We rephrased 1.2 to clarify that the material of the chip is removed all the way to the pickup loop (lines 109-113).

-4.6.1 – Should be "to move the sample".

We rephrased the sentence (line 189).

Tel: 972 3 7384339 :טל: beenalab.biu.ac.il • beena@biu.ac.il

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-Figure 3 legend – "Several manipulation" We changed "manipulation" to "manipulations" (line 248).

#### 6. Additional detail is required:

-2.2 – Is the substrate placed in the chamber after the pre-sputtering step? What are the settings for pre-sputtering?

We added the pre sputtering settings to the protocol, and added step 2.3 to clarify that the substrate is placed in the chamber after the pre-spattering (lines 119-125).

-3.1 – How is the sample loaded on the microscope?

We added a step to the protocol describing this stage (lines 140-141), and added GE Varnish and silver paste to the materials list. We have also added directions for loading the SQUID chip to the microscope (lines 136-139).

-3.5 – How and how far away is the sample moved from the sensor?

The sample is moved using the Z direction stick-slip motion stage, to a distance of 0.5-1mm from the sensor. We added this information to the protocol (line 154).

-3.6 – Please describe the angles. Are they between the sample and the sensor? We rephrased the sentence (lines 156-157), and changed figure 1 (panels c and d) to better explain this.

-3.7 - How close to the sensor?

Sample should be  $1\mu m$  away from the sensor. We added the information to the protocol (line 159).

-3.8 – Please clarify "the first to make contact." Will anything else be making contact with the sample?

We rephrased the sentence so that the meaning is clear (line 163).

-4.2 – How is the magnetic field generated? How does one know the number of vortices? We added an explanation regarding how the magnetic field is applied (lines 171-172). We have also added information to instruction 4.1 and updated the equipment table, in order to further clarify the setup (lines 167-169).

-4.6.2 – How is this monitored? Is there an output to view?

We have changed step 4.6 in order to describe more accurately the approach sequence to the sample (lines 183-201).

-4.7 – How is scanning performed/initiated?

We added a sentence to explain that (lines 203-204), we also added an explanation for achieving a constant height above the sample (lines 205-207).

-4.10 – How greater of a voltage? How is the sensor moved?

We added the information (line 214). We explained the movement of the sensor in 4.7.

7. Branding should be removed from steps 1.1 and 3.4 - attocube We removed the branding (lines 106, 149).

Tel: 972 3 7384339 : של: beenalab.biu.ac.il • beena@biu.ac.il

**Reviewers' comments:** 

Manuscript Summary:

Reviewer #1:

## Faculty of Exact Sciences Department of Physics Dr. Beena Kalisky beenalab.biu.ac.il

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8. Results: Please discuss the results presented in more detail. Two sentences is insufficient. We expanded the introduction to the presented data (lines 226-229). Please advise if this is what you meant.

This is a very strong paper and fits in well with th	is journal on visualization.
Major Concerns: N/A	
Minor Concerns: N/A	
Additional Comments to Authors: N/A	

#### Reviewer #2:

Manuscript Summary:

The authors present an elegant way of manipulating superconducting vortices which may guide future works where removing or controlling vortices in other materials or devices could be useful.

### Major Concerns:

- 1) Mention that these are superconducting vortices right away in the abstract. We clarified that in the abstract (line 49).
- 2) In one place the authors say thousands of vortices, which seems to be unrealistic for manipulating such a large number of vortices.

We added a sentence to clarify that (lines 226-229).

3) The table needs a better formatting: lines, text alignment, another column explaining what each component is for.

The table was a template we filled. However, we resubmitted the table and added a version with better formatting.

Minor Concerns:

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A suggestion only: add a flow diagram to describe better what each component (in the table) function is.

It seems unclear what "by local contact" means in the abstract. Perhaps, "by local elastic deformation" would be more appropriate if that is the underlying mechanism for the vortex manipulation.

The table was a template we filled. We rephrased to "local physical contact" to clarify "local contact" (line 53).

We thank the referee for his\her helpful comments.

Additional Comments to Authors:

N/A

#### Reviewer #3:

Manuscript Summary:

This manuscript describes a method for moving vortices using a scanning SQUID microscope.

Major Concerns:

N/A

Minor Concerns:

Reference 3 is the same as reference 5

We changed reference 5, so it is now the correct one (lines 319-323).

Additional Comments to Authors:

N/A

We thank the referees, and we hope the revised version is suitable for publication.

Sincerely, Eylon Persky

Beena Kalisky's scanning SQUID lab beenalab.biu.ac.il