Focusing of a neutral helium beam below one micron

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Abstract. In 2008 we presented the first images obtained with a new type of matter wave microscope: NEutral Helium Atom MIcroscopy (NEMI). The main features in NEMI are the low energy of the atoms (<0.1 eV) and the fact that they are neutral. This means that fragile and/or insulating samples can be imaged without surface damage and charging effects. The ultimate resolution limit is given by the de Broglie wavelength (about 0.06 nm for a room-temperature beam), but reaching a small focus spot is still a major challenge. The best result previously was about 2 \(\mu\)m. The main result of this paper is the focusing of a helium atom beam to a diameter below 1 \(\mu\)m. A particular challenge for neutral helium microscopy is the optical element for focusing. The most promising option is to manipulate neutral helium via its de Broglie wavelength, which requires optical elements structured to nanometre precision. Here we present an investigation of the helium focusing properties of nanostructured Fresnel zone-plates. Experiments were performed by varying the illuminated area and measuring the corresponding focused spot sizes and focused beam intensities. The results were fitted to a theoretical model. There is a deviation in the efficiency of the larger zone plate, which indicates a distortion in the zone-plate pattern, but nevertheless there is good agreement between model and experiments for the focus size. This together with the demonstration of focusing to below 1 \(\mu\)m is an important step towards nanometre resolution neutral helium microscopy.

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1. Introduction

The importance of microscopy in science and technology can hardly be overstated. Matter wave microscopes, where massive particles are used as the imaging probe, have developed rapidly in recent years with major advancements such as helium ion microscopy [1, 2] and atomic resolution transmission electron microscopy [3]. Common for all commercially available matter wave microscopes is that the imaging probe is a beam of charged particles of quite high energy (typically several keV or more), which may charge the sample if it is not conductive and, due to strong interaction with the matter, it is highly probable that energy transfer from the probe to the atomic/molecular species in the sample can cause sample damage. This makes it difficult to image fragile and/or insulating samples, such as for example polymeric nanostructures, insulating coatings, various biological samples, etc. In some cases, atomic force microscopy or scanning near field optical microscopy can be used instead, but these techniques are not suited for samples with high aspect ratio structures.

In 2008, this group published the first images obtained using a new matter wave microscopy technique (NEMI) where a neutral beam of helium atoms is used as the imaging probe [4]. The low energy of the beam (<0.1 eV) and the fact that the beam is neutral, makes this technique particularly suited for microscopy of fragile and/or insulating samples. Our original images were shadow images obtained in transmission by focusing a helium beam down to about 2 µm with a Fresnel zone plate and scanning the focused beam across a hexagonal copper grid. The image was obtained by recording the signal variation as atoms were transmitted through the grid openings or blocked. Very recently the first neutral helium reflection images were published [5]. The resolution is similar to previous results, but the new reflection images demonstrate beautifully the potential power of neutral helium microscopy as a surface characterization technique.

A crucial point for high-resolution neutral helium microscopy is the optical element. However, since neutral helium in the ground state is not coupled with electric or magnetic fields it is difficult to manipulate it other than via its de Broglie wavelength. The general field of de Broglie matter wave optics, where atoms and molecules are manipulated via their de Broglie wavelength has attracted considerable attention in recent years [6] and has also been a topic for several recent publications in this journal [7–10]. Very recently, the famous Poisson spot experiment for light was carried out for the first time with neutral matter waves, using a µm size circular plate created with electron beam lithography [10, 11]. de Broglie matter
wave optics is characterized by the very small wavelength of matter waves, typically less than 0.1 nm. This puts a very large demand on the optical elements. In some cases light fields are used [12], but this is, in practice, not possible for helium, which means that we are left with material mirrors and lenses. Mirror focusing has been tried on a couple of occasions [8, 13, 14], but the issue of creating the correct mirror shape remains a limiting factor [15], so that the best two-dimensional (2D) focusing obtained with mirrors so far, has only produced a spot size of around 30 µm diameter [8]. Since the energy of the atoms is so low that they do not penetrate materials, the only possible lenses are so-called Fresnel zone plates. A Fresnel zone plate is a special type of axially-symmetric diffraction grating, where the grating period decreases with the distance from the centre. Fresnel zone plates are used extensively, for example, in x-ray optics [16]. A few papers have been published on the application of zone plates in focusing of atoms and molecules [4, 17–19].

Here, we present the best focus of a neutral helium beam that has been obtained so far. We also present a detailed investigation on the focusing properties of two zone plates produced in the group of Professor Günter Schmahl in Göttingen and in the group of Professor Henry I Smith at the Nanostructures Laboratory at MIT (for the rest of the paper they will be referred to as the Göttingen zone plate and the MIT zone plate, respectively). Descriptions of the fabrication steps for each zone plate can be found in [20] and [21]. In both cases the fabrication relies on a combination of electron beam lithography and planar fabrication steps.

The paper begins with a description of the experimental setup used to measure the focusing properties of the zone plates (section 2), then follow the results (section 3) and a description of the theoretical modelling (section 4). The paper finishes with a conclusion.

2. Experimental setup

The experiments presented here were carried out in the molecular beam apparatus popularly known as MaGiE. For a detailed description see [22]. A diagram of the setup is shown in figure 1. A neutral, ground-state helium beam is created by supersonic expansion through a 10 µm-diameter nozzle. For the experiments presented here two different settings were used. The measurements with the Göttingen zone plate were carried out using a beam with an average velocity of \( v = (1129 \pm 3) \text{ m s}^{-1} \) \((E \approx 26.5 \text{ meV})\) and an average wavelength of \((0.0882 \pm 0.0003) \text{ nm}\). The source pressure was 81 bar. For the MIT zone plate the average beam velocity was \( v = (1036 \pm 3) \text{ m s}^{-1} \) \((E \approx 22.3 \text{ meV})\) and an average wavelength of \((0.0961 \pm 0.0003) \text{ nm}\). The source pressure was 110 bar. In both cases the average beam velocity was determined using time of flight (TOF) with a double-slit chopper not shown in figure 1.

The central part of the beam was selected using a micro-skimmer. For the Göttingen zone plate a \((2.5 \pm 0.1)-\mu\text{m}-\text{diameter}\) skimmer was used and for the MIT zone plate a \((1.1 \pm 0.1)-\mu\text{m}-\text{diameter}\) skimmer. The micro-skimmers were made in house using a glass-pipette puller (Narishige PP-830) [23]. The distance between the skimmer and zone plate was \( g = (1528 \pm 5) \text{ mm}\) (see figure 1). To control the illuminated area on the zone plate two collimating apertures with diameters \((300 \pm 5) \text{ µm}\) and \((150 \pm 5) \text{ µm}\) were used (National Aperture Inc.). The apertures were mounted on two linear-motion feedthroughs, which made it possible to move the apertures into and remove them from the beam line. The feedthroughs were placed at distances of \((962 \pm 5)\) and \((802 \pm 5) \text{ mm}\), so that the corresponding illuminated
Figure 1. Diagram showing the experimental setup used in the measurements presented here. The central part of the supersonic expansion beam is selected with a micro-skimmer. Using different collimating apertures the illuminated area on the zone plate can be controlled. The focused spot size is determined by scanning a slit aperture across the focused spot in the image plane. When the helium atoms have passed the slit they are detected in an electron bombardment ionization detector setup. Excluding chromatic aberration the size of the focus is determined by the diameter of the skimmer $d_{sk}$, and the demagnification factor $b/g$.

areas in the plane of the zone plates were $(478 \pm 11) \, \mu m$ and $(288 \pm 11) \, \mu m$, respectively. The MIT zone plate has a diameter of $188 \, \mu m$ and was only tested with full illumination.

To determine the focus spot diameter a $(25 \pm 2 \, \mu m) \times 5 \, mm$ slit aperture (CVI Melles Griot) was mounted on a piezo table (PI model No P-731) and scanned across the focused beam in 0.1 $\mu m$ steps. In a final experiment the slit aperture was replaced with a transmissive sample, a holey carbon film from Quantifoil (R2/1).

3. Results

The zone plates used in this experiment can be seen in figure 2. For more than 100 zones the optical properties of a zone plate can be treated similar to a thin refraction lens [24]. The focal length $f$ of a zone plate depends on the wavelength $\lambda$ of the incident beam and can be determined using the formula [16]:

$$f = \frac{r^2}{N \cdot \lambda},$$

where $r$ is the radius of the zone plate and $N$ the number of zones.

For a wavelength of 0.088 nm the Göttingen zone plate with $N = 2700$ and $r = 270 \, \mu m$ has a focal length of 306 mm. For a wavelength of 0.096 nm the MIT zone plate with $N = 189$ and $r = 96 \, \mu m$ has a focal length of 486 mm. For $g = (1528 \pm 5)$ mm (see figure 1) this corresponds to image distances of $b = (383 \pm 2)$ mm and $b = (713 \pm 4)$ mm, respectively. The optimum image distances were determined experimentally by measuring the focus diameters for different values of $b$ and they were found to be $b = (378 \pm 5)$ mm and $b = (712 \pm 5)$ mm, respectively, in good agreement with the expected values.
Figure 2. Scanning electron microscope images of the Göttingen zone plate [20] and the MIT zone plate [21] used in these experiments. The Göttingen zone plate (top images (a) and (b)) is 540 µm in diameter with a middle stop 162 µm in diameter (blocking the zeroth order) and 2700 zones including the blocked middle stop zones. In (a) the innermost zones are visible with the middle stop. The radial support rods keeping the blocked zones in place are also visible. In (b) the outermost zone with a width of about 48 nm can be seen. The MIT zone plate (bottom images (c) and (d)) is 188 µm in diameter with a 60 µm diameter middle stop and has 189 zones including the blocked middle zones. The middle stop is visible to the right in (c). In (d) the outermost zone with a nominal width of 323 nm can be seen. For an ideal zone plate there should be a 1 : 1 ratio between the blocked and open zones. Both zone plates have slightly wider open zones presumably due to over-exposure or over-etching during fabrication.

Figure 3 shows a typical scan of the full beam profile after the beam has passed through the zone plate, including the zeroth order. The image has been obtained by scanning the beam with a (25 ± 2) µm wide slit using the Göttingen zone plate fully illuminated. The width of the zeroth order corresponds to the size of the zone plate as discussed in [4].

The focused spot sizes were evaluated following the procedure originally introduced in [18] and further developed in [25]: A (25 ± 2) µm wide slit is scanned across the focused spot in
Figure 3. Slit aperture scan for a full beam profile unfocused (filled circles) and focused (open circles) with the Göttingen zone plate using an illumination area diameter of \((478 \pm 11) \mu\text{m}\). The full beam profile scan without the zone plate in the beam line (filled circles) is plotted in the graph with an offset of 500 counts for better visibility in the figure. Both scans were performed with a \((25 \pm 2) \mu\text{m}\) wide slit moving in \(6.5 \mu\text{m}\) steps. Depicted is the measured intensity (count rate per second) versus the slit position. The dash-dotted line and the dashed line represent the fit using a simple diffraction model \([21]\) with a ratio between blocked and open zones of 0.5 (theoretically expected) and 0.42 \(\pm\) 0.01 (best fit), respectively. The zone plate transmits \((3.8 \pm 0.6)\%\) into the focus; expected was 7.7%.

Sub-micron steps (0.1 \(\mu\text{m}\)). As the slit moves across the spot, the measured intensity rises and based on this rise the spot size can be determined by a deconvolution of the measurements with a slit function. Figure 4 shows a typical slit scan. To avoid contributions from temperature fluctuations in the laboratory the measurements were performed by taking fast scans 0.1 \(\mu\text{m}\) steps, 0.5 s per measuring point. Each slit scan took about 60 s. To reduce the effect of temperature fluctuations several measurements were taken for each spot diameter (7–20 scans). Obtaining these results was experimentally very challenging, pushing alignment, temperature control and overall instrument stability to its absolute limit.

In a final experiment the slit was replaced by a carbon film with a hexagonal pattern of 2 \(\mu\text{m}\) circular holes with a periodicity of 3 \(\mu\text{m}\). This sample was used to create the first sub-micron resolution images obtained with helium microscopy. The images are shown in figure 6.

4. Analyzing the results

The two crucial parameters for the zone plates are: (i) Do we obtain the expected focused spot size and (ii) Do we obtain the expected signal intensity in the focused spot—in other words, does the zone plate have the expected efficiency. These two issues are treated in the next two subsections.
Figure 4. Slit aperture scan across a sub-micron focused spot. The scan was performed with 0.1 µm steps. Depicted is the measured intensity (count rate per second) versus the slit position on the piezo table weighted with the expected statistical fluctuation of the count rate (\(\sqrt{N}\)) represented by the error bars. The solid line represents the fit of the data with a Gaussian error function. The measurement was taken using the Göttingen zone plate.

4.1. Focused spot size

Figure 5 presents the focused spot diameters obtained with the two zone plates. The focused spot diameter is plotted versus the diameter of the illuminated area on the zone plates. The results present averages of several measurements (at least 7 per result) as discussed in the previous section. The lines in the diagram present the theoretical model used to predict the focused spot size \(d_\text{th}\). As can be seen there is good agreement between the theoretical model and the experimental results.

The theoretical model is explained in the following: the spread in wavelength for a supersonic atomic beam can be determined from the speed ratio \(S\) (a measure for the velocity distribution) as [26]:

\[
S = 2 \cdot \sqrt{\ln 2} \cdot \frac{v}{\Delta v} \simeq 2 \sqrt{\ln(2)} \frac{\lambda}{\Delta \lambda}.
\]  

(2)

Here, \(v\) is the mean velocity, \(\Delta v\) is the full-width at half-maximum (FWHM) of the velocity distribution and \(\lambda\) and \(\Delta \lambda\) the wavelength and FWHM of the wavelength distribution. The FWHM for a point source (point spread function) \(d_p\) can now be determined using simple geometrical arrangements; see figure 7 and [16]. We obtain

\[
d_p = \frac{r_i}{\sqrt{2\ln(2)}} \simeq 2\sqrt{\ln(2)} \frac{r}{S},
\]

(3)

where \(r_i\) is the radius of the illuminated area of the zone plate.
Figure 5. Focused spot diameters measured with three different size illuminated areas on the zone plates (diameters: $\Omega_1 = 478 \, \mu m$, $\Omega_2 = 288 \, \mu m$ and $\Omega_3 = 188 \, \mu m$). The solid line shows the theoretically calculated focused spot diameters for a beam with speed ratio $S = 286$ and skimmer diameter $d_{sk} = 2.5 \, \mu m$. The dashed line shows the theoretically calculated focused spot diameters for a beam with speed ratio $S = 408$ and skimmer diameter $d_{sk} = 1.1 \, \mu m$. The filled squares are theoretical values including the experimental uncertainties in wavelength, illuminated area and distances $(g, b)$.

The speed ratio $S$ can in principle be determined experimentally using a TOF measurement. The TOF signal is a convolution of the actual velocity distribution of the beam with the chopper slit and the detector function. For the experiments presented here, the source pressures were rather high and therefore the velocity distribution rather narrow. This meant that the width of the chopper-slit transmission curve was rather large in comparison with the velocity distribution and hence we could not determine the velocity distribution with sufficient accuracy using the standard deconvolution procedure [27, 28]. Therefore, we chose to use theoretically determined parameters for the speed distributions calculated using the method of moments with the Lennard-Jones potential [29–31]. Previous experiments show that there is very good agreement between theory and experiment in the pressure and temperature range used for these experiments [31].

In reality the source is not a single point. The extended size of the source has to be included to determine the theoretical focus diameter. Previous experiments show that the spatial intensity distribution in a supersonic expansion source (the so-called virtual source) can be approximated well with two Gaussian functions [32] (a single Gaussian function does not describe the tails of the distribution properly). The width of the two Gaussian functions depends on the nozzle diameter, source pressure and source temperature. On the other hand, the skimmers used here are smaller than the true virtual source and the beam can be considered as emanating from the skimmer. Therefore, a reasonable approximation, which will simplify the following analysis,
Figure 6. 2D transmission scans of a holey carbon foil QUANTIFOIL® (R2/1). The holes are 2 \( \mu \)m in diameter with a period of 3 \( \mu \)m. All pictures were taken with the Göttingen zone plate. (a) Image size 15 \( \times \) 15 \( \mu \)m\(^2\), focused spot diameter < 2 \( \mu \)m, step size 300 nm, 2 s per step; (b) image size 10 \( \times \) 10 \( \mu \)m\(^2\), focused spot diameter < 2 \( \mu \)m, step size 100 nm, 2 s per step; (c) image size 7 \( \times \) 7 \( \mu \)m\(^2\), focused spot \( \leq \) 1 \( \mu \)m, step size 100 nm, 2 s per step; (d) image size 4 \( \times \) 4 \( \mu \)m\(^2\), focused spot diameter \( \leq \) 1 \( \mu \)m, step size 100 nm, 1 s per step. Images (c) and (d) are distorted because of thermal fluctuations, which becomes a factor due to the longer scan times caused by the small step size and the low intensities (a smaller area of the zone plate is illuminated to get better focus and hence the signal is smaller).

is to assume a Gaussian distribution with FWHM corresponding to the skimmer diameter \( d_{Sk} \). The FWHM of the geometrical image size of the source is then simply \( d_{op} = (b/g) \cdot d_{Sk} \) (see figure 1). The final theoretical focus diameter \( d_{th} \) can then be obtained as a convolution of two Gaussian functions: the geometrical image and the point spread function:

\[ d_{th} = \sqrt{(d_p)^2 + (d_{op})^2} \]  

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4.2. Zone-plate efficiency

Looking at figure 4 one notices that the overall count rate in the focus is quite low (only a few hundred counts). In fact, the number of helium atoms arriving at the detector is much higher, but the very low efficiency of the present-day electron-bombardment detectors (less than $10^{-5}$) makes it appear so low. An analysis of the transmissivity and efficiency of zone plates produced at MIT has been published in [21]. The results show that the transmissivity of the MIT zone plates is very close to what would be theoretically expected. No results have been published so far on the efficiency of the Göttingen zone plate. In figure 3, a scan of the complete beam profile and the beam profile focused with the Göttingen zone plate is shown. The expected efficiency for the Göttingen zone plate is 7.7% (corresponds to the dash-dotted line in figure 3). This number stems from the theoretical efficiency for a zone plate with 0.5 open fraction (ratio between open zones and total area), with the additional area of the middle stop and the support pattern subtracted [33]. We see that the efficiency is significantly less than expected, even though the width of the beam focus has the expected value. The efficiency obtained experimentally for the Göttingen zone plate is $(3.8 \pm 0.6)\%$. Here efficiency refers to the ratio of the measured intensity in the first-order focus divided by the intensity incident on the illuminated part of the zone plate without the area blocked by the middle stop. The analysis also shows that the open fraction is significantly less than expected, $0.42 \pm 0.01$ (compared to 0.5). This will lead to a reduction of the intensity transmitted into the first-order focus. The electron microscopy images in figure 2 show that the outermost blocked zones appear to be too narrow, which one would expect would lead to an increase in the open fraction. A possible explanation for the low open fraction could be the van der Waals interaction between the zone plate and the helium atoms. Van der Waals interaction between a nanostructured grating and molecular beams is described among others in [34]. We have tested this hypothesis using the theoretical approach described in our recent publication in this journal [10], and found that the only effect is a small reduction in the effective grating width, as also noted in [34], which was not enough to explain a decrease in the open fraction of almost 20%. The large deviation must therefore mostly be due to a deviation of the zone-plate pattern from its ideal. In figure 3, it can be noted that there is a

Fig. 7. This drawing illustrates the geometrical relation between the change in focal length $\Delta f$ due to a change in wavelength and the resulting point spread function $d_p$. $r_i$ denotes the radius of the illuminated area of the zone plate.
surplus of diffuse intensity within about 50 µm of the sharp first-order focus when compared to the diffraction model (dashed line). This can only be explained by a distortion of the zone-plate pattern.

5. Conclusion

Figure 5 shows that sub-micron focusing of a neutral helium beam is possible. Further, figure 5 shows very good agreement between the theoretically predicted focus and the experimentally measured values for various zone plate diameters. Even for the largest illuminated areas there is good agreement. This is very encouraging. There is a deviation in efficiency for the Göttingen zone plate, which we attribute to a distortion in the zone-plate pattern, the focus diameter, however, is still as expected. We thus conclude that the zone plates are patterned to a precision close to specifications paving the way for nanometre resolution neutral helium microscopy. The slit scan measurement shown in figure 4 illustrates one of the problems that we are still facing. The count rate appears very low—only a few hundred counts. In fact, the number of helium atoms arriving at the detector is much higher, but the very low efficiency of the present-day electron-bombardment detectors (less than $10^{-5}$) makes it appear so low. Several groups are currently exploring the possibility of improving the detector efficiency with various means [35–41], but so far no breakthrough has occurred. The other crucial issue is thermal drift. This should be possible to solve quite easily, by designing an instrument of smaller dimensions, comparable to a helium ion or a scanning electron microscope, which can be kept in a temperature controlled environment.

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