# **Lawrence Berkeley National Laboratory**

**Lawrence Berkeley National Laboratory** 

### **Title**

Improved Spatial Resolution for Reflection Mode Infrared Microscopy

# **Permalink**

https://escholarship.org/uc/item/61v0h27h

#### **Author**

Bechtel, Hans A.

# **Publication Date**

2009-12-16

Peer reviewed

# Improved Spatial Resolution for Reflection Mode Infrared Microscopy

Hans A. Bechtel, Michael C. Martin, T. E. May, and Philippe Lerch

<sup>1</sup>Advanced Light Source Division, Lawrence Berkeley National Laboratory, 1 Cyclotron Road, Berkeley, CA 94720, USA

<sup>2</sup>Canadian Light Source Inc., University of Saskatchewan, 101 Perimeter Road, Saskatoon, Saskatchewan S7N 0X4, Canada

#### **Abstract**

Standard commercial infrared microscopes operating in reflection mode use a mirror to direct the reflected light from the sample to the detector. This mirror blocks about half of the incident light, however, and thus degrades the spatial resolution by reducing the numerical aperture of the objective. Here, we replace the mirror with a 50% beamsplitter to allow full illumination of the objective and retain a way to direct the reflected light to the detector. The improved spatial resolution is demonstrated using two different microscopes capable of diffraction-limited resolution: the first microscope is coupled to a synchrotron source and utilizes a single point detector, whereas the second microscope has a standard blackbody source and uses a focal plane array (FPA) detector.

<sup>&</sup>lt;sup>3</sup>Swiss Light Source, Paul Scherrer Institut, 5232 Villigen, Switzerland

<sup>&</sup>lt;sup>a)</sup> Author to whom correspondence should be addressed. Electronic mail: MCMartin@lbl.gov. Tel.: 510-495-2231. FAX: 510-495-2067

Infrared microscopes are typically designed to be used with standard blackbody sources. Because these sources suffer from poor brightness, spatial resolution is controlled by geometrical apertures that are typically limited by signal-to-noise ratios to 20 µm or larger. In the midinfrared, these aperture sizes are far from the diffraction limit. Consequently, most designers choose to maximize the throughput of the system rather than achieve the best possible spatial resolution at the diffraction limit. Technological advances in infrared sources and detectors, namely synchrotron infrared radiation<sup>1-9</sup> and focal plane arrays, <sup>10</sup> however, have made diffraction-limited spatial resolutions possible by either increasing the source brightness<sup>11</sup> or by multiplexing data acquisition. Here, we discuss a simple modification to commercial infrared microscopes that allows the user to take full advantage of the spatial resolution achievable by these new sources and detectors.

Infrared microscopes operate in either transmission or reflection mode, with the choice primarily depending on the optical properties of the sample and/or the substrate. In transmission mode (Fig. 1a), an objective focuses the infrared radiation onto the sample and a second objective or condenser collects the transmitted radiation before directing it to the detector. In the standard reflection mode (Fig. 1b), light that is reflected off the sample is collected by the same objective that focuses the infrared light. The light is then directed to the detector by a "sluice" mirror, which is removed during transmission mode measurements. The mirror, however, blocks a portion of the incident light and effectively shadows half of the objective's secondary mirror. Because the spot size for a diffraction-limited system is determined by the wavelength of light and the numerical aperture of the objective, this half-illumination degrades the spatial resolution of the microscope.

The spot size in a diffraction-limited system can be modeled by the point spread function (PSF) of the objective. Figures 2a-c show the simulated PSF<sup>12</sup> of a common Schwarzschild objective used in mid-infrared microscopy (32x, NA 0.65) with full illumination of the secondary mirror, as is the case with transmission mode. The PSF of a Schwarzschild objective has a narrower zero-order peak and larger first- and higher order diffraction maxima than the familiar Airy pattern of a typical visible microscope objective because the secondary mirror of the Schwarzschild objective obscures a portion of the central aperture. While a standard optic has about 85% of the total sensitivity in the zero-order peak, the Schwarzschild objective has only 50% in the central region, leading to possible loss of image contrast when used for scanning microscopy. The effects of the first- and higher order diffraction maxima on resolving power, however, can be reduced by operating the microscope in a confocal arrangement, as demonstrated by Carr. 4

Figures 2d-f show the PSF of the same 32x Schwarzschild objective when the secondary mirror is half-illuminated, as is the case with the standard reflection mode. The reduction in numerical aperture creates a spot size that is more than double in one dimension than in the other dimension. This elliptical spot can distort images created by scanning microscopy, particularly at longer wavelengths where the effects of diffraction are more obvious. An alternative method of reflection that achieves the same resolution as in transmission mode replaces the "sluice" mirror with a beamsplitter (Fig. 1c). Although this design limits the throughput to 50% of the standard reflection mode, the maximal spatial resolution for the microscope is achieved because the secondary mirror is fully illuminated.

In order to experimentally verify the improved spatial resolution of the beamsplitter reflection mode, infrared measurements were performed at the Advanced Light Source (ALS)

and the Swiss Light Source (SLS). For measurements at the ALS, synchrotron infrared light at beamline 1.4.3 was directed through a Nicolet Magna 760 FTIR interferometer bench with a KBr beamsplitter and a Spectra Tech Nic-Plan IR microscope (32x objective, NA=0.65) before being detected with a mercury cadmium telluride (MCT-A) detector. Sample images were acquired with an automated microscope stage (Prior ProScan II) capable of step sizes as small as 0.1 µm. For reflection measurements in beamsplitter mode, the Nic-Plan microscope was modified to accommodate a 50:50 CaF<sub>2</sub> beamsplitter (ISP Optics) at the position of the "sluice" mirror. In this case, the CaF<sub>2</sub> beamsplitter limits the spectral range to wavelengths shorter than 10 µm, but other beamsplitter substrates (e.g. KBr) could be used to obtain spectra of the entire mid-infrared spectral region if necessary. The modifications to the microscope allow easy switching between the standard and beamsplitter reflection modes. For measurements at the SLS, infrared light from a globar source was directed through a Bruker Vertex 70 FTIR interferometer with a KBr beamsplitter and coupled into a Bruker Hyperion 3000 microscope with a 128 element FPA detector. For reflection measurements in beamsplitter mode, the sluice mirror was replaced with a metal mesh that acted as a beamsplitter in the mid-IR spectral region.

Three types of resolution tests were performed in order to demonstrate the improved spatial resolution of the beamsplitter reflection mode. In the first test, an 8  $\mu$ m Ti dot on a Si substrate was imaged using the standard and beamsplitter reflection modes (Fig. 3) with a synchrotron source. At  $\lambda=2$   $\mu$ m, the two images are nearly identical because the diffraction pattern for both modes of reflection is smaller than the test sample. At longer wavelengths, however, the standard reflection mode images are clearly distorted in one dimension and appear elliptical (oriented at approximately 45° because of the optical design of the microscope). The

images from the beamsplitter reflection mode, on the other hand, remain symmetrical and relatively unchanged with increasing wavelength, in agreement with the PSF simulations.

The second resolution test was a step-edge (or knife-edge) test with a synchrotron source using a high resolution USAF 1951 3-Bar Resolving Test Chart (MILSTD- 150A, section 5.1.1.7) from Applied Image Inc. (Rochester,NY, USA). The sample was stepped in 0.5  $\mu$ m increments from a position on the absorbing/transmitting glass pattern to a position on the reflective metal coating along a line oriented at 45 degrees, corresponding to the degraded resolution in the standard reflection mode. By acquiring a spectrum at each point, a profile of the reflectivity as a function of position and wavelength was obtained. The first derivative of the profile and the FWHM of a Gaussian fit are comparable to the PSF and resolution of the system, respectively. Figure 4 shows the first derivative of the step profiles for the standard and beamsplitter reflection modes at  $\lambda=8~\mu m$  obtained without any apertures. The standard reflection mode profile is clearly wider than the zero and first order diffraction maxima of the beamsplitter reflection mode profile. A simple linear fit to the data between  $\lambda=2~\mu m$  and  $\lambda=8~\mu m$  yields  $(0.73\pm0.05)\lambda$  resolution for the beamsplitter reflection mode and  $(1.83\pm0.05)\lambda$  resolution for the standard reflection mode.

The first two tests demonstrate the improved diffraction-limited performance of the beamsplitter reflection mode when using a synchrotron source, which is 100-1000 times brighter than a globar source. Indeed, the ability to achieve diffraction-limited performance with high signal to noise ratios is the primary reason that infrared spectromicroscopy beamlines exist worldwide. Recent technological advances in mid-infrared focal plane arrays, however, have enabled diffraction limited performance with globar sources, although synchrotron sources still retain their superiority in terms of ultimate spatial resolution and signal to noise ratios. <sup>14</sup>

Therefore, in the final resolution test, the beamsplitter reflection mode is examined with a microscope equipped with a globar source and a focal plane array detector. As seen in Figure 5, the beamsplitter reflection mode provides superior resolving power over the standard reflection mode.

By illuminating the entire secondary mirror, the beamsplitter reflection mode achieves the same symmetric illumination and therefore the same diffraction-limited spatial resolution obtained in transmission mode. This improved spatial resolution over the standard reflection mode, however, comes at the expense of infrared light throughput. If the measurements are shotnoise limited, the factor of two reduction in throughput should reduce the signal-to-noise ratio by  $\sqrt{2}$ . In measurements at the ALS, we find the signal-to-noise ratio of the beamsplitter reflection mode to be only slightly worse than the standard reflection mode because shot-noise is usually not the limiting noise source. An rms noise value of <0.05% is routinely achieved on a 100% line on gold (128 scans, 4 cm<sup>-1</sup> resolution) for the beamsplitter mode, allowing both good sensitivity and high spatial resolution for infrared measurements of small samples or regions of samples.

# Acknowledgements

The authors thank G. L. Carr for help with Zemax simulations. The Advanced Light Source is supported by the Director, Office of Science, Office of Basic Energy Sciences, of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231.

#### References

J. A. Reffner, P. A. Martoglio, and G. P. Williams, Rev. Sci. Instrum. 66, 1298 (1995).

G. L. Carr, J. A. Reffner, and G. P. Williams, *Rev. Sci. Instrum.* **66**, 1490 (1995).

M. C. Martin and W. R. McKinney, *Proc. Mater. Res. Soc.* **524**, 1 (1998).

G. L. Carr, Rev. Sci. Instrum. 72, 1613 (2001).

- <sup>5</sup> M. C. Martin and W. R. McKinney, *Ferroelectrics* **249**, 1 (2001).
- <sup>6</sup> P. Dumas and M. J. Tobin, *Spectr. Eur.* **15**, 17 (2003).
- <sup>7</sup> L. M. Miller and P. Dumas, *Biochim Biophys Acta* **1758**, 846 (2006).
- E. Levenson, P. Lerch, and M. C. Martin, J. Synchrotron Rad. 15, 323 (2008).
- E. Levenson, P. Lerch, and M. C. Martin, *Infrared Phys. Techn.* **51**, 413 (2008).
- E. N. Lewis, P. J. Treado, R. C. Reeder, G. M. Story, A. E. Dowrey, C. Marcott, and I. W. Levin, *Anal. Chem.* 67, 3377 (1995).
- W. D. Duncan and G. P. Williams, *Appl. Opt.* **22**, 2914 (1983).
- <sup>12</sup> Zemax (Zemax Development Corporation).
- J. C. Russ, *The Image Processing Handbook*. (CRS Press, Boca Raton, 2002).
- E. Levenson, P. Lerch, and M. C. Martin, *Infrared Phys. Techn.* **49**, 45 (2006).

### **Figure Captions**

**Figure 1.** (Color online) Schematic diagram of three different modes of infrared microscopy: a) transmission, b) reflection with the standard mirror design, and c) reflection with the beamsplitter design.

**Figure 2.** (Color online) a) Surface plot, b) image plot, and c) horizontal cross section of the point spread function of a 32x Schwarzschild objective with full illumination of the secondary mirror at  $\lambda = 6 \mu m$ , which is the case for the transmission and beamsplitter reflection modes. d) Surface plot, e) image plot, and f) horizontal cross section of the point spread function of the same objective with half-illumination of the secondary mirror, which is the case for the standard reflection mode.

**Figure 3.** (Color online) Images of an 8 μm Ti dot on a Si substrate at different wavelengths for standard and beamsplitter reflection modes using a synchrotron source and a single point detector.

Figure 4. (Color online) a) Gaussian fits and b) PSF simulations (solid lines) superimposed on the first derivatives of step edge profiles at  $\lambda = 8 \, \mu m$  for the standard reflection mode (black circles) and the beamsplitter reflection mode (blue triangles) using a synchrotron source and a single point detector.

**Figure 5.** (Color online) Infrared images of a patterned sample obtained with a) standard reflection mode and c) beamsplitter reflection mode using a globar source and a focal plane array. b) Optical image of the sample.

Figure 1

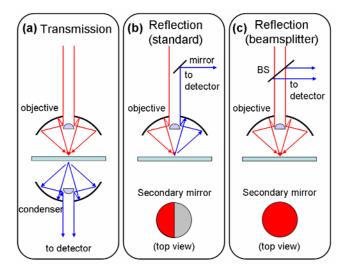


Figure 2

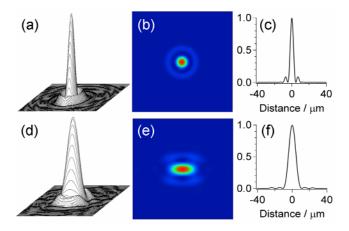


Figure 3

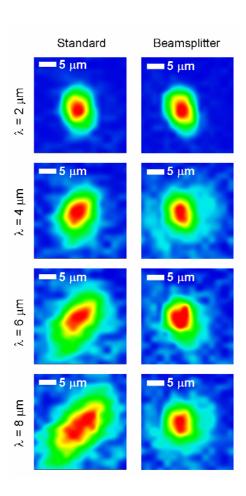


Figure 4

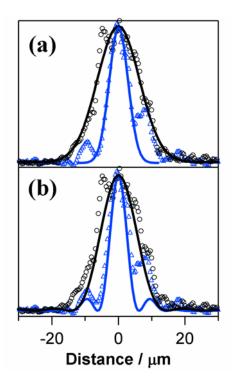


Figure 5

