Fire damage on carbon fiber materials characterized by THz waves

Nicholas Karpowicz[a], David Dawes[b], Mark J. Perry[b], and X.-C. Zhang[a]

[a]Center for Terahertz Research
Department of Physics, Applied Physics and Astronomy
Rensselaer Polytechnic Institute
110 8th Street, Troy, NY 12180
zhangxc@rpi.edu

[b]Battelle Memorial Institute
505 King Drive, Columbus, OH 43201
perrymj@battelle.org

Abstract
We apply THz imaging technology to evaluate fire damage to a variety of carbon fiber composite samples. The majority of carbon fiber materials have polarization-dependent reflectivities in the THz frequency range, and we show how the polarization dependence changes versus the burn damage level. Additionally, time domain information acquired through a THz time-domain spectroscopy (TDS) system provides further information with which to characterize the damage. The technology is discussed in terms of non-destructive testing applications to the defense and aerospace industries.

Keywords: Terahertz / Non-destructive testing / Non-destructive evaluation / composites

1. Introduction

Imaging in the THz frequency range has been a subject receiving considerable attention in recent years, with hopes of access to new methods in diverse subjects ranging from medicine to manufacturing to security. The impetus for this interest springs from three aspects of radiation in this part of the electromagnetic spectrum: 1) The preponderance of spectroscopic signatures of large molecules lying within this range, 2) that imaging systems based on THz waves can achieve resolutions easily amenable to human interpretation (i.e., the detail acquired does not require leaps of imagination on the part of the operator in order to understand the information therein) and 3) the transparency of many commonly-used materials within this range (including plastics, fabrics and, to a degree, ceramics). The types of THz radiation used for imaging falls into two categories, pulsed[1] and continuous-wave[2-6] (CW). A pulsed system sends a short (typically less than one picosecond) transient onto or through a sample, and coherently records the resulting waveform, which can be analyzed in both time and frequency domains. CW imaging works with a single frequency, which in the absence of rapid tuning may inhibit spectroscopic imaging, but has the advantages that it is simpler to reduce into a compact imager and can be simpler to operate since the signal does not need to be sought in the time domain.

As one of the major applications of THz imaging is in the field of non-destructive testing, we have applied it to an area of critical need: the testing of aerospace materials. Composite materials such as carbon fiber are widely used in this industry. The nature of their use requires technologies that are able to differentiate between safe and unsafe materials, either due to manufacturing tolerance or damage acquired while in use. In this paper, we discuss the applicability of terahertz (THz) imaging systems to this purpose, focusing on graphite fiber composite materials. We have previously demonstrated the use of both

Terahertz for Military and Security Applications IV, edited by
Dwight L. Woolard, R. Jennifer Hwu, Mark J. Rosker, and James O. Jensen,
Proc. of SPIE Vol. 6212, 62120G, (2006) · 0277-786X/06/$15 · doi: 10.1117/12.665852

continuous-wave and pulsed THz imaging systems for the evaluation of space shuttle insulating foam[6], which is an ideal target for such systems due to its low absorption and index of refraction in this spectral range. Carbon fiber-based materials present more of a challenge, as the fibers are conductive and therefore exhibit a high THz reflectivity. As a result, our measurements are uniformly performed in a reflection imaging geometry, which does have the advantage of more accurately simulating the type of measurement that could be performed in a real-world setting.

The material used in this study falls somewhat outside of the range of typical THz imaging subjects as it is not highly transparent in bulk form. The conducting fibers will reflect or absorb the incoming signal either immediately at the surface or within a few sample layers, depending on the polarization of the incoming waves. The usefulness of this type of information depends on the type of damage one is attempting to detect and the specifics of the structure being studied. We will show that in some cases the information provided by a THz image gives a more complete outline of the damaged area than what is apparent in a visible light image, and that time-domain processing with data from a pulsed THz system can reveal changes to the structure of the surface of the material.

2. Experimental Setups

Two systems were used to study the samples in these measurements: a THz time-domain spectroscopy (TDS) system and a CW THz imager operating at 0.6 THz, both of which have been described elsewhere.[7] In short, the THz TDS system begins with a Ti:Sapphire oscillator (Spectra-Physics Mai Tai), which produces pulses of 800 nm central wavelength, 80 fs duration, and 80 MHz repetition rate with an average power of 800 mW. The laser beam is split into pump and probe beams, the former being incident on a p-type InAs wafer, which produces the THz pulses and the latter is recombined with the THz signal after it is focused onto and returned from the sample. The THz and probe pulses travel collinearly through a ZnTe crystal, which serves as an electro-optic sensor of the full amplitude and phase of the THz pulse. The system is shown schematically in Fig. 1.

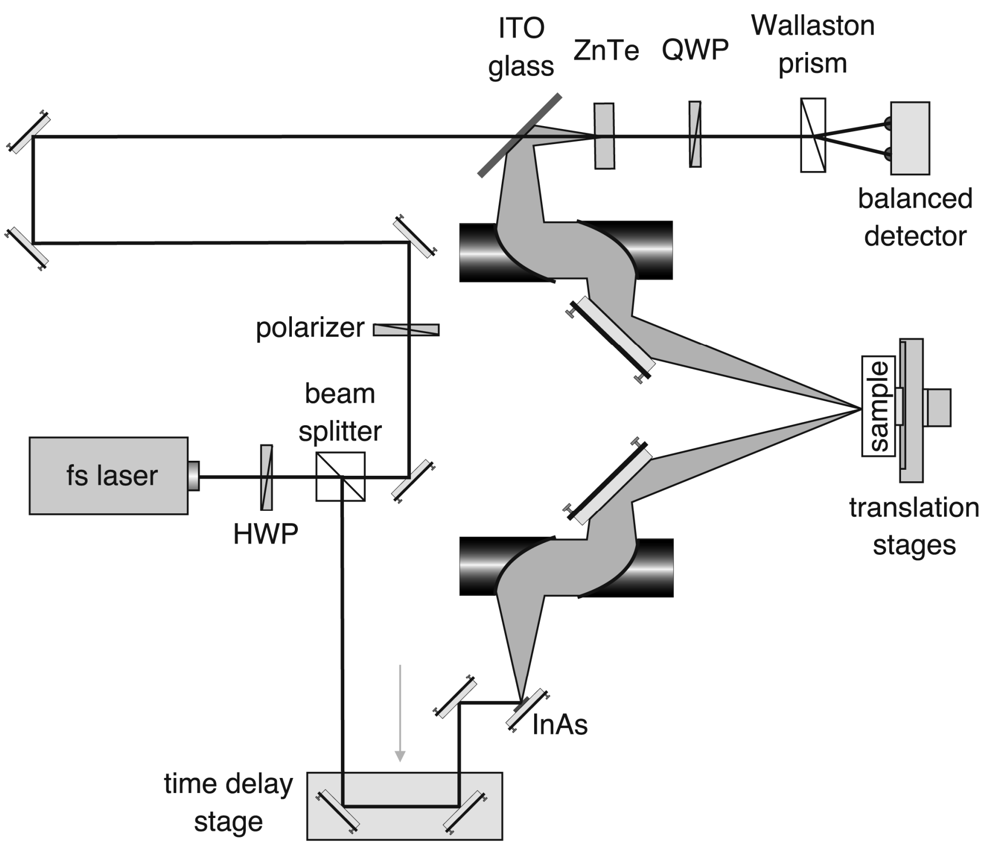

Fig. 1: Schematic diagram of the THz TDS system used in the experiment.

The CW system, a diagram of which is shown in Fig. 2, consists of a Gunn diode oscillator operating at 100 GHz, which is then frequency doubled and tripled to 0.6 THz. The beam is focused by a hyperbolic polyethylene lens to a 500 μm spot, collected by the same lens after being reflected by the sample and is measured by a Golay cell. The sample is mounted on a pair of translation stages and moved to produce the image.

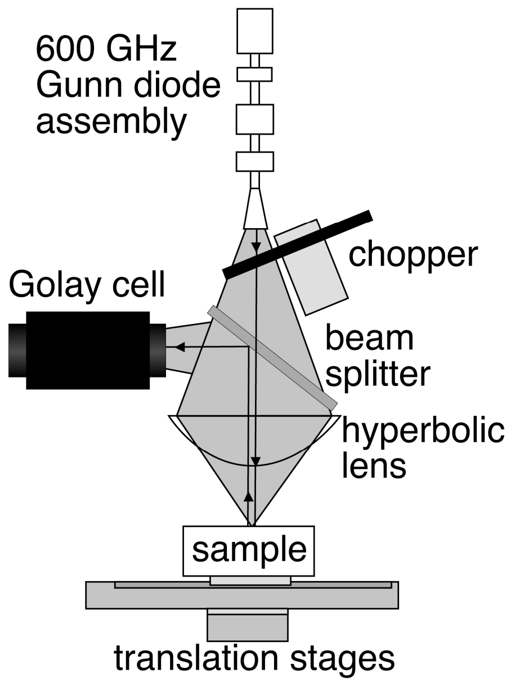

Fig. 2: Schematic diagram of the continuous-wave THz imaging system used in the experiment.

3. Continuous-wave imaging

The CW images reveal features that are important for application: damaged areas show a strong change in reflectivity, and the reflectivity is highly dependent on the polarization of the incoming radiation. The latter is quite similar to the function of a wire grid polarizer, which reflects waves whose electric field is parallel to the grid and allows those whose field is perpendicular to pass. Thus, the image with parallel electric field shows mainly information about the topmost surface, while the perpendicular field image penetrates several layers further. The reflectivity can be described as

$$R = R_G \cos^2\theta_P + R_B \qquad (1)$$

where R_G describes the polarization-sensitive reflectivity of the fiber grid, and R_B describes the background reflectivity of the sample, which arises from the fact that the fibers are not entirely unidirectional and are densely spaced in a dielectric resin. These two parameters can be extracted via measurements at orthogonal polarizations. Once measured, R_G and R_B give indications of the severity and nature of the damage to the sample. If there is a significant reduction of R_G after a sample is damaged, this indicates that there is damage to the fibers, e.g. through reorientation or a chemical change resulting in lower conductivity such as residual surface char from the matrix. A reduction of R_B can attributed to damage to the resin, conductivity loss, or increased surface roughness.

In Fig. 3, we show the results of imaging a severely damaged sample and applying this characterization framework. The four images shown are the optical image, as well as images of the reflectivity with the electric field parallel to the fibers ($R_{||}$), R_G, and R_B (which is equivalent to the reflectivity perpendicular to the fibers). It can be seen that the areas of change to R_G and R_B differ, indicating that the nature of the damage is not uniform across the damaged region. It can also be seen that the contrast in the THz images is much higher than that of the optical image (although, the resolution is lower). One may also observe that when the polarization is perpendicular to the fiber direction, more structure is visible, due to increased penetration of the beam into the sample.

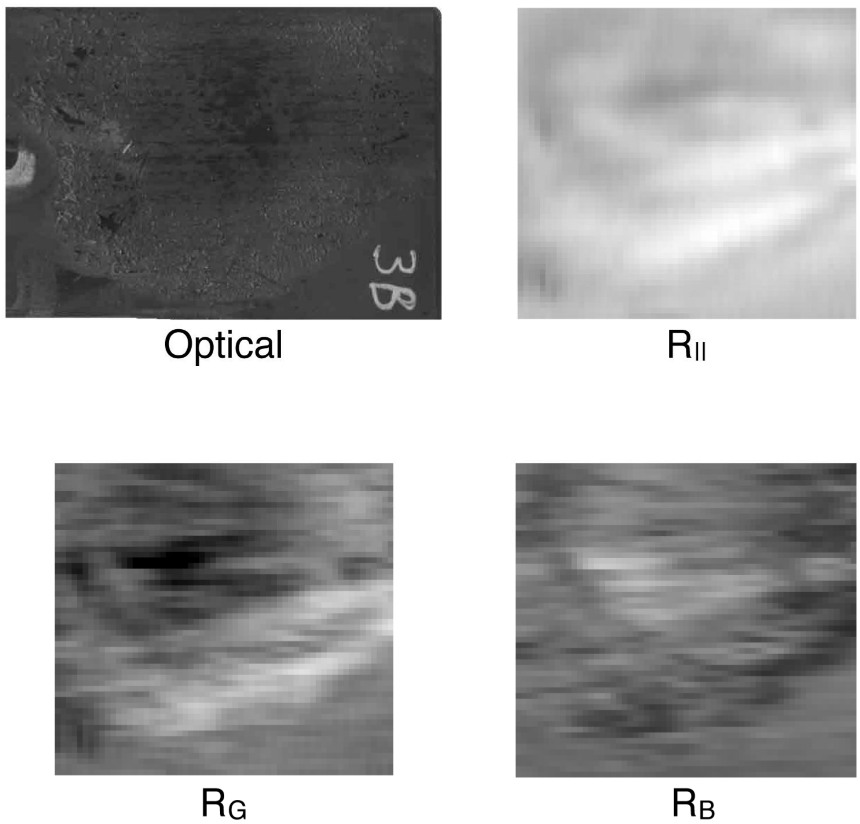

Fig. 3. Imaging results for a sample of carbon fiber composite with severe burn damage (the imaged area is 28 x 31 mm). R|| is formed with the THz wave polarized such that the electric field is parallel to the fibers of the material, resulting in the highest reflectivity. R_G and R_B are images of the parameters of Equation 1, where R_G is the polarization-sensitive reflectivity of the grid of wires and R_B is the background reflectivity. R_B is equal to the reflectivity obtained when the polarization of the THz wave causes the electric field to be perpendicular to the fibers.

An important consideration for a non-destructive evaluation technique is the rate of false calls. This is mitigated in this imaging modality by the fact that the images provided are easily recognizable to human vision. To test the case of minor damage that does not have an effect on the strength of the material, but it still apparent in a visual inspection of the material, we imaged a lightly burned sample of material, which is shown in Fig. 4. It can be seen that the scorch mark that is apparent in the optical image does not appear in the THz image since the burning did not affect the underlying structure. That the severity of the damage in terms of effect on the physical strength of the material is correlated with the apparent effect in the THz images was confirmed in a separate work[8].

In order to apply this CW imaging technique to real-world applications, there are several important considerations. First is the geometry of the material being studied. In these examples, a flat sample of material is used under normal incidence conditions. If the material is curved, or if normal incidence is impossible, a more complicated system will be required in order to ensure that the beam enters the detector. One must also know the fiber orientation, which for many carbon fiber materials is quite obvious, but if the polarization is not aligned precisely parallel and perpendicular to the fiber direction, the extraction of R_G and R_B becomes more difficult (and impossible at a 45^o polarization angle).

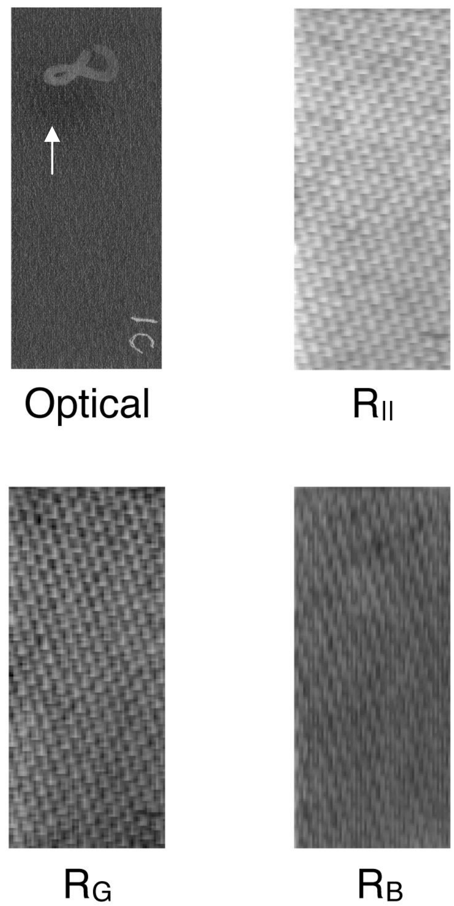

Fig. 4. Results of applying CW imaging (in an identical matter to that of Fig. 3) to a sample with insignificant burn damage (the imaged area measures 80 x 35 mm). It can be seen that the scorch mark visible in the optical image (indicated by the white arrow) does not appear in the THz images. It is noteworthy that this sample has a more complicated fiber structure than that of Fig. 3, which is apparent in the THz image, but not in the optical image since the fibers are covered by an optically-opaque dielectric layer.

4. THz Time-Domain Spectroscopy Measurements

Measurements with a coherently detected THz pulse through THz time-domain spectroscopy (TDS) yield additional information about the material. Since the measurement is time-resolved, it is possible to extract the presence and depth of multiple reflections from the surface. By deconvolving the waveform reflected by a suspect location with a waveform from an undamaged location, it is possible to precisely locate the reflection events at the surfaces in the time domain[9-13]. This information can yield a description of important types of material deformation such as delamination, wherein the multiple layers of the sample become separated from one another. We further described this processing in another work.

An example of THz TDS waveforms from a damaged and undamaged sample surface are shown in Fig. 5. There is a clear double-peak structure in the waveform reflected by the damaged surface, indicating delamination. This shows that time-domain THz measurements are able to add a third dimension to imaging of carbon fiber composites, further characterizing the nature of the damage.

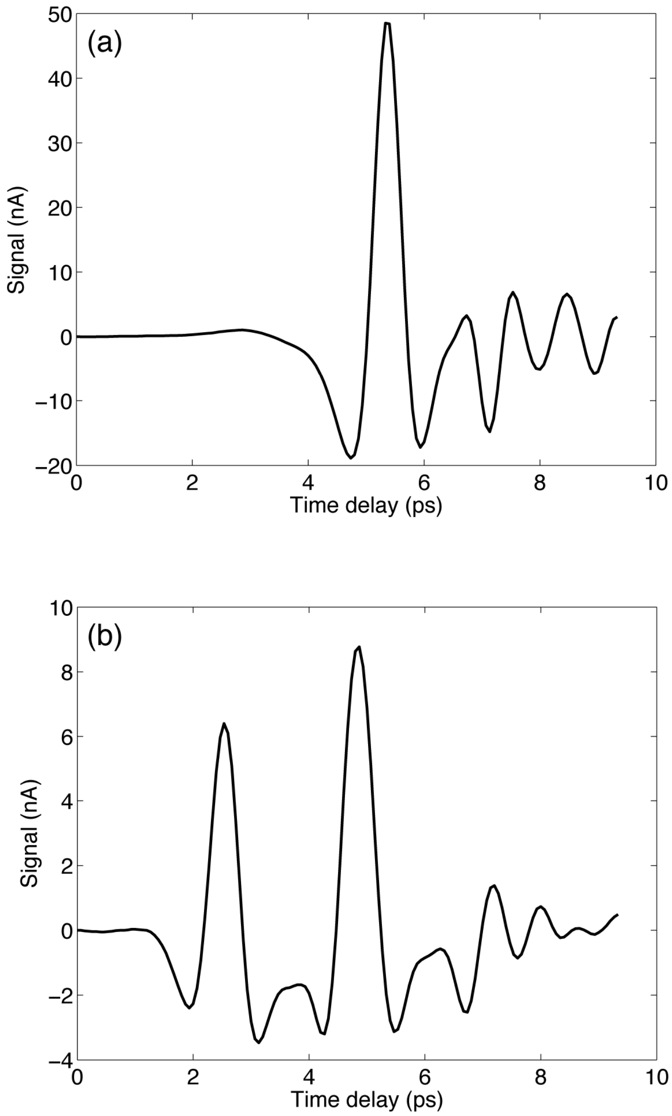

Fig. 5. Time-domain waveforms taken from the material shown in Fig. 3, (a) undamaged and (b) damaged. The splitting of the peak in the time domain indicates the presence of multiple reflecting surfaces. In the case of this material, the large separation between the peaks (in this geometry, the 2.5 ps separation is equivalent to a 130 μm separation between the reflecting surfaces), indicates delamination.

Since TDS measurements are resolved in the time domain, it adds another dimension of sensitivity to the material geometry, however. If the distance to the target changes, the position of the pulse in the time domain will shift, and if the detection window is too small, disappear. One can simply increase the size of the time window, but this increases acquisition time, which may not be acceptable for a given application.

Real-world application of these techniques requires speed as well as specificity. The examples shown in this paper utilize a raster scanning technique to form the images, wherein a THz beam is focused and scanned across the sample, with the detected reflected signal being recorded point by point. Such an imaging modality is time-consuming and most-likely will be inappropriate for large-scale applications or testing where time is an issue. For these cases, "camera-like" operation would be much preferred. Steps have been taken in this direction, for both pulsed and CW THz techniques.

At its most basic level, camera functionality requires an array of detectors and focusing elements capable of forming an image upon it. For CW THz systems, detector array options include commercially available arrays of pyroelectric detectors, which unfortunately suffer from high noise-equivalent power, and microbolometer arrays, which have been shown to produce usable, real-time imaging combined with quantum cascade lasers[14]. Combined with appropriate optics, which can include systems made of reflective optics (e.g. parabolic mirrors) or lenses made of THz-transparent materials such as polyethylene, Teflon or Picarin glass, such detector arrays can be readily adapted to this application.

Focal plane imaging has been demonstrated with pulsed THz systems, as well. A commonly used method is to utilize a large electro-optic crystal, such as one made of ZnTe, and an expanded optical probe beam[15,16]. The THz image projected onto the crystal will spatially modulate the polarization of the probe beam, which can be detected using polarization optics and a CCD. Such a system requires a strong THz pulse (such as those generated using an amplified Ti:Sapphire laser), which would currently prohibit field applications. Additionally, the EO crystal should be extremely uniform and have very little strain, as crystal variation and stress have a strong effect on the crystal birefringence. While it is possible to meet these requirements, such a crystal can be prohibitively expensive, especially at large sizes.

5. Conclusion

THz imaging and spectroscopy are promising solutions to the problem of identifying evaluating damage to carbon fiber composite materials. While additional progress in THz technology will be required for some applications, currently existing tools are able to provide useful and quantifiable information regarding the extent and severity of damage. Such techniques could potentially increase safety and efficiency in the defense and aerospace industries.

Acknowledgements:

The authors thank Xu Xie, Hua Zhong, Michael Shur and Nezih Pala for technical assistance. This material is based upon work supported by the National Science Foundation under Grant No. 0333314.

References:

1. B. B. Hu and M. C. Nuss, “Imaging with terahertz waves,” *Opt. Lett.* **20**, 1716 (1995).

2. T.S. Hartwick, D. T. Hodges, D. H. Barker, and F. B. Foote, “Far Infrared Imagery,” *Appl. Opt.* **15**, 1919 (1976).

3. T. Kleine-Ostmann, P. Knobloch, M. Koch, S. Hoffmann, M. Breede, M. Hofmann, G. Hein, K. Pierz, M. Sperling and K. Donhuijsen, “Continuous-wave THz imaging,” *Electronics Letters,* **37** 1461 (2001).

4. K. Siebert, H. Quast, R. Leonhardt, T. Löffler, M. Thomson, T. Bauer, and H. G. Roskos, “Continuous-wave all-optoelectronic terahertz imaging,” *Appl. Phys. Lett.* **80** 3003 (2002).

5. A. Dobroiu, M. Yamashita, Y. N. Ohshima, Y. Morita, C. Otani, and K. Kawase, “Terahertz imaging system based on a backward-wave oscillator,” *Applied Optics* **43** 5637 (2004).

6. N. Karpowicz, H. Zhong, C. Zhang, K.-I Lin, J.-S. Hwang, J. Xu and X.-C. Zhang, “Compact continuous-wave subterahertz system for inspection applications,” *Appl. Phys. Lett.* vol. **86**, 054105, 2005.

7. B. Ferguson and X.-C. Zhang, “Materials for terahertz science and technology,” *Nature Materials* **1**, 26 (2002).

8. Nicholas Karpowicz, David Dawes, Mark J. Perry, and X.-C. Zhang, "Terahertz characterization of fire damage to carbon fiber materials," *Appl. Phys. Lett.* (submitted).

9. R. A. Cheville and D. Grischkowsky, *Appl. Phys. Lett.* “Time domain terahertz impulse ranging studies,” **67,** 1960 (1995).

10. D. M. Mittleman, S. Hunsche, L. Boivin, and M. C. Nuss, “T-ray tomography,” *Opt. Lett.* **22**, 904 (1997).

11. T. Dorney, W. W. Symes, R. G. Baraniuk and D. Mittleman, “Terahertz multistatic reflection imaging,” *J. Opt. Soc. Am. A*, **19**, 1432 (2002).

12. D. M. Mittleman, M. Gupta, R. Neelamani, R. G. Baraniuk, J. V. Rudd, and M. Koch, “Recent advances in terahertz imaging,” *Appl. Phys. B,* **68,** 1085 (1999).

13. K. McClatchey, M. T. Reiten, and R. A. Cheville, “Time resolved synthetic aperture terahertz impulse imaging,” *Appl. Phys. Lett.*, **79**, 4485 (2001).

14. A. W. Lee and Q. Hu, "Real-time, continuous-wave terahertz imaging by use of a microbolometer focal-plane array," *Opt. Lett.* **30**, 2563-2565 (2005).

15. Jiang Z. P. and Zhang X.-C., “Terahertz imaging via electrooptic effect,” *IEEE Trans. Microw. Theory Tech*. **47,** 2644 (1999).

16. Jiang, Z. and Zhang, X.-C., “Single-shot spatiotemporal terahertz field imaging.” Opt. Lett. **23**, 1114 (1998).