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Terahertz Self-Referencing Technology for Characterizing XNO® Material-Based Battery Anodes: Advancements in Performance Evaluation and Analysis

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ABSTRACT

Terahertz technology represents a cutting-edge advancement in non-destructive testing, facilitating rapid and inline inspection of high-value coatings across various industries including automotive, pharmaceuticals, semiconductors, and aerospace. Among its recent applications is the assessment of battery electrodes. In this study, five mixed niobium oxide (XNO®) anodes, supplied by Echion Technologies Ltd, both calendered and uncalendered, underwent THz measurements. The results unveiled a thickness range of 25 μm to 45 μm, closely aligning with micrometer values at 99.16%. Furthermore, the observation of a 1.6 THz absorption peak in XNO® reflection underscores the potential of terahertz technology in material research applications.

Keywords: Terahertz, non-destructive testing, electrode coating inspection, material research

Introduction

Terahertz technology stands out as a versatile tool for non-destructive sensing, offering rapid and reliable assessment of thin layered films. In the realm of battery electrode assessment, where parameters like density, thickness, and conductivity are paramount, terahertz sensors present a novel solution. Maintaining uniform coating thickness is crucial to ensuring electrode performance and minimizing degradation rates, while density optimization balances energy storage capacity with power requirements. Moreover, enhancing coating conductivity is essential for maximizing capacity, particularly under high discharge rates. Leveraging terahertz self-referencing technology enables simultaneous measurement of these critical parameters for the first time.

When a terahertz pulse interacts with a battery electrode, it initiates a complex interplay: part of the radiation reflects from the surface, while the remainder passes through, engaging with the current collector and producing a secondary reflection. The initial reflection, stemming from the electrode surface, correlates with the coating material's complex refractive index, wherein density exhibits a direct correlation. The time lapse between these reflections is product of the real part of the refractive index and the layer thickness. Meanwhile, the second reflection provides insights into the material's conductivity, elucidating the imaginary component of the refractive index. Thus, terahertz technology empowers the determination of both thickness and complex dielectric properties without necessitating prior knowledge of material composition [1-3].

Result and discussion

XNO®-based batteries exhibit superior rate performance compared to graphite-based batteries, particularly evident in lithiation and delithiation rate tests conducted at low temperatures. Despite increased impedance and charge transfer resistance observed at lower temperatures, XNO®-based batteries maintain exceptional performance, enabling rapid charging and discharging even in challenging conditions, highlighting their advantage over graphite-based alternatives [4-5].

(a)

Figure 1: (a) terahertz Self-referencing Reflection Sensor developed at TeraView Ltd

Five XNO® anodes provided by Echion Technologies, measured with terahertz Self-referencing (SR) reflection sensor [6] developed at TeraView Ltd, Cambridge, UK (see Figure 1). The SR sensor is engineered and manufactured for deployment in industrial settings where regular referencing may pose challenges or be inconvenient. It operates by automatically acquiring an internal reference during each measurement. This sensor was connected to a TeraPulse Lx time-domain terahertz spectrometer and equipped with an 18.5 mm focal length lens in a throw-catch configuration. The angle of incidence for the incident beam is 15.6 deg and the polarization is set to s-reflection.

The thickness of the anodes ranged from 23 µm to 43 µm, closely aligning with micrometer measurements at 99.16%. These anodes were both calendered and uncalendered. The measured terahertz refractive index correlated with a density range of 1.56 to 2.81 g/cm³. As expected, uncalendered samples exhibited greater thickness but lower density, while calendered samples, subjected to pressure during calendering, were thinner and denser.

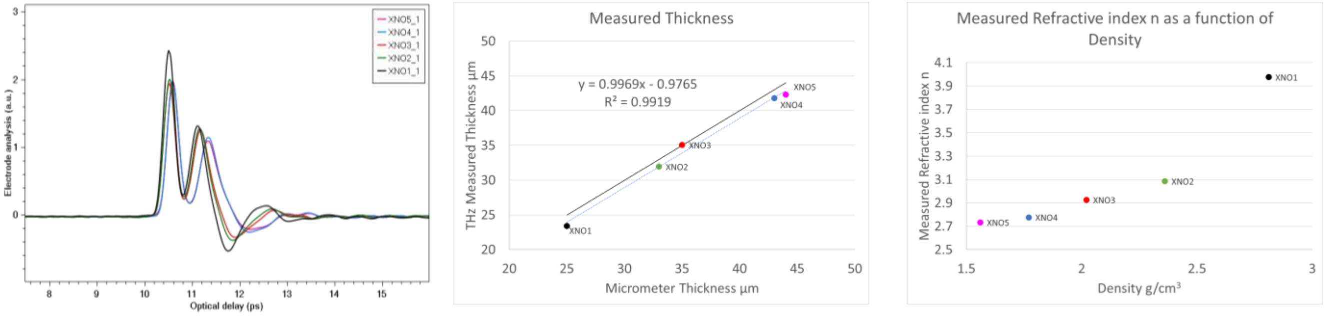
Table 1: Terahertz Thickness and Micrometer Readings of Five Anodes, Along with Density and Refractive Index Data

Sample	Micrometer (µm)	THz Thickness (µm)	Density (g/cm^3)	Terahertz refractive index
XNO$_5$	44	42.3	1.56	2.73
XNO$_4$	43	41.8	1.77	2.78
XNO$_3$	35	35.1	2.02	2.93
XNO$_2$	33	31.9	2.36	3.09
XNO$_1$	25	23.4	2.81	3.98

Figure 2(a) illustrates the waveform measured from anode 1 to anode 5, all made from XNO® material and analyzed in TeraPulse software version 0.15.179. To remove edge effects in the waveform we use Tukey apodisation function set to 10% of the length of optical time delay. Thickness measurements from each anode were compared with micrometer readings, and the comparison showed less than a 1% error, as depicted in Figure 2(b). The data are detailed in Table 1. The electrode analysis developed at TeraView enables refractive index measurements of materials without prior knowledge of the material. The refractive index is reported in Table 1 and correlated to density in Figure 2(c). The surface reflection of the anodes, as mentioned, correlates with the density of the coating. As depicted in Figure 2(c), XNO$_1$ (black-line graph) exhibits the highest surface reflection compared to the other samples, while simultaneously possessing the highest density.

This analysis demonstrates the efficacy of THz measurements in characterizing the thickness and refractive index of XNO® anodes. The correlation between terahertz refractive index and density provides valuable insights into the structural properties of the anodes, particularly in distinguishing between calendered and uncalendered samples. These findings underscore the utility of THz technology in material characterization and suggest its potential for quality control and optimization in manufacturing processes.

Further investigation into the terahertz reflection from XNO® material unveils an absorption peak at 1.6 THz. This discovery not only highlights the remarkable capabilities of terahertz technology in material research but also sheds light on the underlying physics governing the behavior of XNO® material. Specifically, the appearance of the absorption peak at 1.6 THz suggests a significant interaction with the material's Fermi energy level.

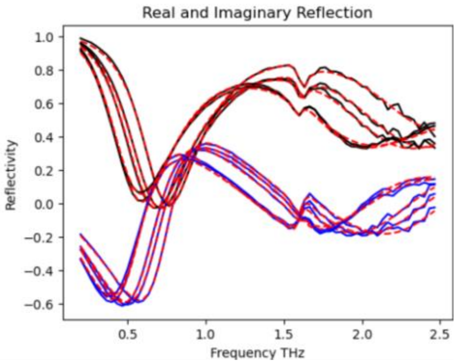


(a) (b) (c)

Figure 2: (a) Deconvolved waveform (b) Terahertz thickness measurement compared with measured thickness (c) Terahertz refractive index correlated to the density.

Analyzing the real and imaginary components of the reflection from XNO® anodes provides deeper insights into their optical properties. These components, shown in Figure 3, denoted by black and blue curves respectively, are compared with a theoretical model employing a Lorentzian oscillator (represented by a red dotted line). The agreement between experimental data and the model indicates the presence of characteristic features within the XNO® material's response to terahertz radiation.

These findings deepen our understanding of XNO® material's optical properties and its interaction with terahertz radiation. Moreover, they underscore the potential applications of terahertz spectroscopy in elucidating the vibrational dynamics of materials, offering avenues for further exploration in fields ranging from materials science to condensed matter physics.



(a)

Figure 3: Terahertz spectrum reveals absorption peak of XNO® material occurring at 1.6 THz.

Conclusion

In conclusion, our comprehensive analysis of THz measurements on calendered and uncalendered XNO® anodes has provided valuable insights into their structural and optical properties. Through precise correlation of terahertz refractive index with density, we have elucidated distinct characteristics between the two types of samples, highlighting the effectiveness of THz technology in material characterization. Furthermore, the identification of an absorption peak at 1.6 THz suggests significant interactions within the XNO® material, shedding light on its underlying physics. By employing advanced analysis techniques, Lorentzian oscillator model, we have further elucidated the optical behavior of XNO® anodes, paving the way for future applications in the field of materials science. These findings not only deepen our understanding of XNO® material but also underscore the potential of terahertz spectroscopy in advancing nondestructive testing research and development efforts.

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