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Modelling THz TDS Spectra of Powder Compacts for Analysis of Pharmaceutical Oral Solid Dosages

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***Abstract*—THz TDS is a vital tool in many industrial sectors, however commonly analyzed samples are often produced as powders or granular materials; these morphologies have been demonstrated to produce anomalous spectral features which appear at unpredictable locations and can hamper spectral analysis. Here we systematically study the effects of sample morphology on the appearance of anomalous spectral features and present a physical mechanism to describe their behavior. Two-part powder compacts of PTFE and Borofloat glass are prepared at a range of microsphere sizes and concentrations for TDS measurement. Further, a computational model utilizing ray-tracing through heterogeneous material is developed, which is able to accurately predict the anomalous spectral features. By attributing a physical mechanism to this phenomenon, we are able to predict their occurrence, and utilize their presence to deduce sample morphology.**

I. INTRODUCTION

THE presence of spurious spectral features in powder samples has been reported by various researchers in a number of materials [1-5], which occur unpredictably, often obscuring known resonance features, and making material identification challenging. These spectral features are known to be dependent on sample morphology, however to date there has not been a valid physical mechanism reported which fully explains the phenomenon. Here we utilize extensive experimental measurements of PTFE and Borofloat powder compacts with varying morphology. Further, we develop a ray-tracing simulation method to model the propagation of THz pulses through heterogeneous samples. The simulation is capable of generating spectra which match closely to experimental measurements and provides a compelling explanation as to the physical mechanism which generates these anomalous spectral features.

II. EXPERIMENTAL RESULTS

Powder compacts consisting of PTFE with Borofloat microspheres were prepared with a range of microsphere concentrations and sizes. The samples were compacted at 400 MPa and allowed one week for relaxation of any elastic stress. The fabricated samples were circular disks of 9 mm diameter and 1 mm thickness. This preparation method has been confirmed to produce powder compacts of negligible porosity and sufficient tensile strength for repeated measurements. The samples are measured in transmission mode THz-TDS (Teraflash Pro, Toptica) in purged dry air, allowing for a high dynamic range up to 4.5 THz. Control samples of homogeneous PTFE compressed powder and a Borofloat wafer were also measured. The TDS spectra were analyzed to extract effective refractive index and loss coefficient.

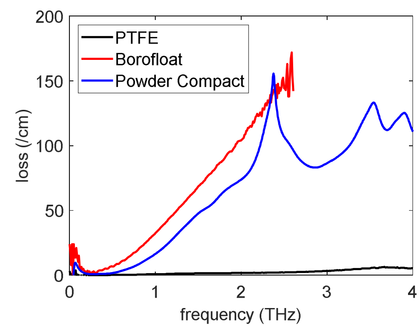

Fig. 1. Experimental measurements of homogeneous PTFE and Borofloat present featureless loss spectra, however when combined in a powder compact a sharp spectral feature appears.

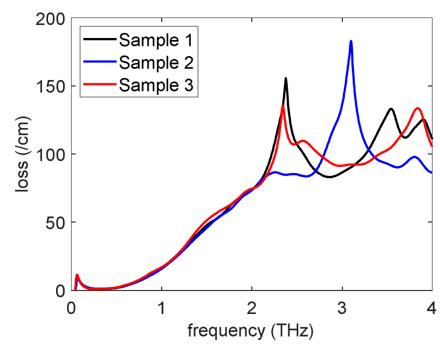

Fig. 2. Experimental measurements of powder compacts prepared to identical morphology (~98 μm at 20 % v/v concentration) displaying dramatic differences in loss spectra.

Figure 1 presents the loss spectra for homogeneous PTFE and Borofloat samples, overlaid with the loss spectra for a powder compact (~98 μm diameter microspheres at 20 % v/v concentration). The homogeneous materials display rising, featureless, loss spectra over the observed frequency range, however the Borofloat spectra is invalid over 2.6 THz due to low signal-to-noise ratio. Unlike the homogeneous materials, the powder compact displays a sharp resonance feature close to 2.2 THz, followed by two smaller peaks at 3.5 and 3.8 THz. A wide range of microsphere sizes and concentrations were analyzed. It was found that the frequencies of spectral peaks appear to shift as the size and concentration of microspheres is altered.

Curiously, the spectral features also change dramatically even when the sample morphology is kept constant. **Figure 2** presents loss spectra for three samples produced to identical morphological preparation, with microspheres of ~98 µm diameter at 20 % v/v concentration. Although they have identical morphology, the spectra reveal clear visible differences.

III. Ray-Tracing Simulation

To elucidate the physical mechanism of the spurious spectral features observed in experiments, a bespoke ray-tracing simulation was designed in MATLAB. A schematic of the simulation geometry is provided in **Figure 3**. The simulation consists of a 2D sample containing a multitude of randomly located circular scattering centers, representing Borofloat microspheres in a PTFE matrix. Rays are initialized at the top surface of the sample with a randomised incidence angle and position in the *x* axis. The rays are traced through the sample in straight lines until incident upon a scattering centre. If a scattering event is detected, the ray's angle will be altered. Utilizing the complex refractive index of the PTFE and Borofloat materials, the propagation time and intensity of each ray is tracked during its transit. Rays which emerge at the lower face of the sample are considered as being transmitted, and their exit time and intensity are logged. A total of one million rays are traced through each sample. The exit times of each ray are binned, with the contribution to the bin determined by their intensity, to produce a 'temporal profile'.

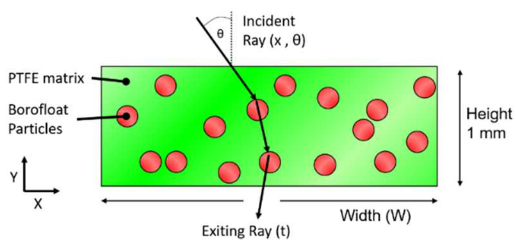

Fig. 3. Schematic of the simulated sample geometry, displaying randomly located scattering centers which alter the trajectory of incident rays.

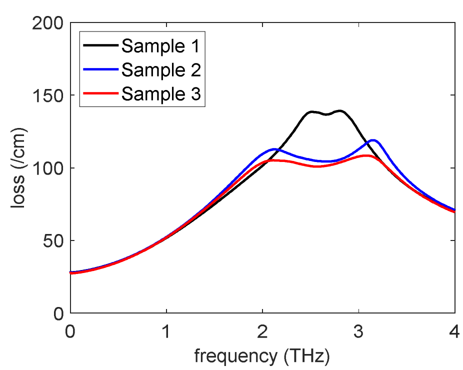

Fig. 4. Simulated loss spectra for three samples of identical parameters (scattering centre size and concentration) displaying sample-dependent spectral features.

A Fourier transform is used to obtain the frequency domain of the sample's response, where the magnitude of the Fourier transform is converted into a component which is analogous to experimentally measured loss coefficient. **Figure 4** plots the 'loss coefficient' obtained from the ray-tracing simulation for scattering centers of 98 µm at 20 % concentration, simulated for three samples. Each simulated sample contains identical parameters, however the randomized locations of the scattering centers are altered for each sample. The spectra reveal the presence of several sharp peaks located between 2 – 3.5 THz, which are of similar location to those seen in experimental measurements. Further, like the experimental measurements, the simulation produces loss spectra which are substantially different even when sample parameters (size and concentration of scattering centers) are held constant.

IV. Summary

Spurious features in the loss spectra of powder compacts are observed experimentally, and appear to change in number-of-peaks and frequency-location unpredictably. A ray tracing simulation is designed to model propagation through heterogeneous material to gain insight into this effect. The simulation is able to produce spectra which match closely to experimental measurements. The physical mechanism of this effect is elucidated by analyzing the time-domain of the ray-tracing simulation, which explains not only the presence of the spurious features, but also how samples of identical morphology can produce varying numbers of peaks at various frequency locations. By providing a physical mechanism to these spurious features, we are able to predict their behavior, or use their presence to infer details of sample morphology, thereby extending the utility of THz-TDS for heterogeneous samples such as pharmaceuticals and agriculture.

V. Grant Acknowledgments

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VI. Data Availability

All the data associated with this paper are openly available from Zenodo [http://doi.org/10.5281/zenodo.14930787] under a Creative Commons Attribution (CC BY) license.

VII. References

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