Material Characterization using Microwaves

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Abstract:
The present work proposes designs and implements a novel microwave based material characterization technique. By connecting a klystron based microwave source to a transmitter antenna, a turn table rotates the sample to be characterized, placed in the radiation field of the antenna, by 360 degrees. A receiver antenna then collects the scattered radiation, and the distribution of intensity for various angles is recorded using power meter. Nonlinear analysis techniques such as spectrum, phase portrait, polar plot and Lyapunov exponents are then used to characterize the crystalline/amorphous nature of the sample. The techniques are validated for nanostructured samples of Titanium Oxide as well as Zinc Oxide based thin films. It is hoped that these preliminary results will lay the foundation steps towards microwave based material characterization, with the significant advantage of lesser health hazard compared with X-Ray based techniques.

Keywords: Material Characterization, Microwave, Scattering, Lyapunov Exponent, Nonlinear Analysis

1. Introduction

The advancement of nanoscience and nanotechnology have without doubt opened the doors for useful applications of a variety of micro and nano-structured materials in diverse fields of fields ranging from biochemistry to electrical engineering [1-2]. Subsequently, this advancement has necessitated the development of characterization techniques such as Electron Microscopy and X-Ray Diffraction in order to establish the structure, texture and other physical properties of the fabricated materials [3-4].

The present work proposes a characterization technique based on microwaves. Specifically, by focusing X-Band microwave generated using Klystron sources on material samples placed on a turn-table, the scattered radiation is collected by a receiving antenna, and the intensity distribution for all 360 degrees of rotation is studied. Nonlinear analysis techniques such as phase portraits and Lyapunov Exponents are used in conjunction with polar plots and spectra to understand the material structure from the obtained distributions. It is hoped that these preliminary results will lay the foundation steps towards microwave based material characterization, with the significant advantage of lesser health hazard compared with X-Ray based techniques.

2. Design and Results

The proposed design is illustrated as a block schematic in Fig. 1. The setup essentially consists of a microwave source connected to a transmitting horn antenna. The sample to be characterized is placed in the radiation path of the antenna, and is controlled using a microcontroller controlled turn-table, which rotates the sample 360 degrees. The scattered radiation in the direction of transmission is collected by a receiver horn antenna, which is then connected to a power meter, measuring the received power intensity, and is plotted as a polar plot of intensity vs angle.

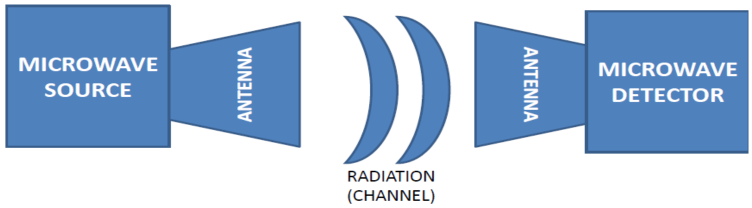

Figure 1 Block Diagram of the Microwave Material Characterization Setup

Reflex klystrons are used as the microwave source. A reflex klystron is essentially a single cavity klystron based oscillation device, where microwave oscillations are produced by virtue of converting the velocity modulation of electrons to current modulation in the repeller space with a typical X-band (8-12GHz) signal of around 3W power [5]. Crystal detector connected to a power meter is used as the detector. The experimental setup is shown in Fig. 2.

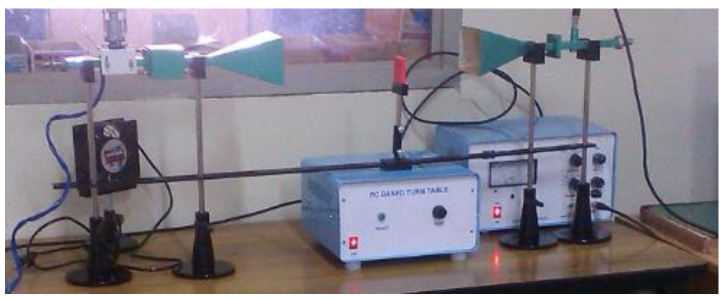

Figure 2 Photo of the Microwave Material Characterization Setup

In order to perform material characterization, the following techniques are used:

1. Spectrum: Plotted using the Fast Fourier Transform, the Spectrum highlights periodicities and other repetitive elements in the time series data, obtained by converting the polar data into Cartesian coordinates [6].
2. Phase Portrait: This is a plot of time derivative of the signal in terms of the signal, illustrating the phase space dynamics and qualitatively serving as a tool to assess sensitivity and ergodicity. The detection of ornamental and rich patterns in a phase portrait is a clear indicator of the presence of chaos underlying the scattering dynamics [7].
3. Polar Plot: Plotted as a function of magnitude and phase, the polar plot helps to understand the component-wise and collective phase distributions in the signal, distinguishing between the noise floor and chaotic components present therein [6].
4. Largest Lyapunov Exponent: This is a measure of a system's sensitive dependence on initial conditions. In the present work, Rosenstein's algorithm is used to compute the Lyapunov Exponents λ_i from the voltage waveform, where the sensitive dependence is characterized by the divergence samples $d_j(i)$ between nearest trajectories represented by i given as follows, C_j being a normalization constant [8-9]:

$$d_j(i) = C_j e^{\lambda_1 (i\Delta t)}; d(t) = C e^{\lambda_1 t} \qquad (1)$$

Firstly, a 3cmx3cm sample of Titanium (Ti) substrate on which a 5um thick film of 20nm grain size Rutile-Titanium Dioxide (TiO_2) is deposited is considered. The nonlinear analysis is performed for this sample with both and oxide parts facing the transmitter, and the results are plotted in Fig. 3 and 4.

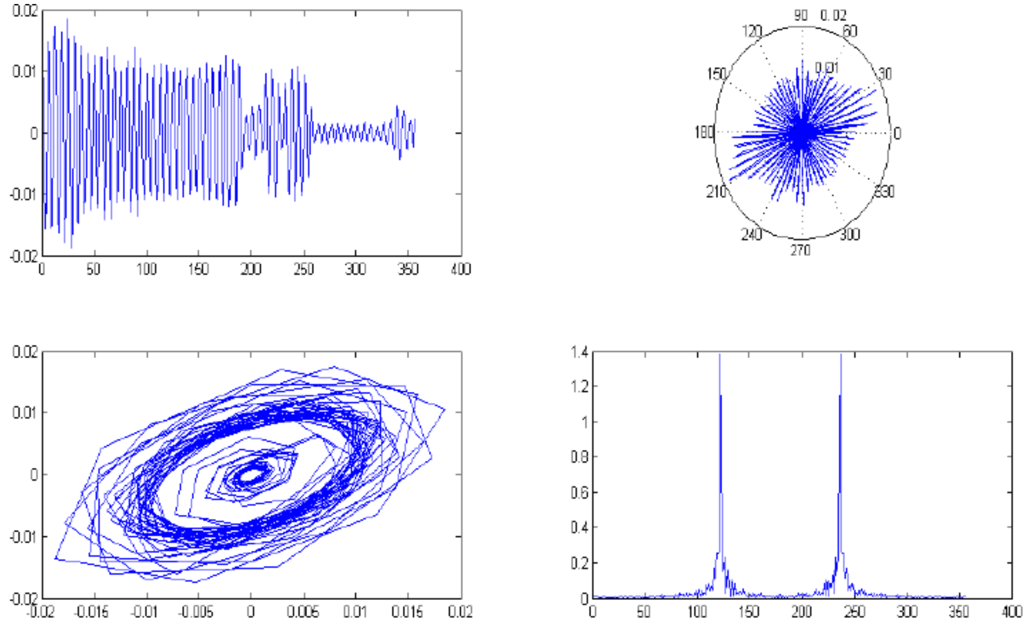

Figure 3 Waveform, Polar Plot, Phase Portrait and Spectrum of Ti/TiO_2 sample, Ti facing Transmitter

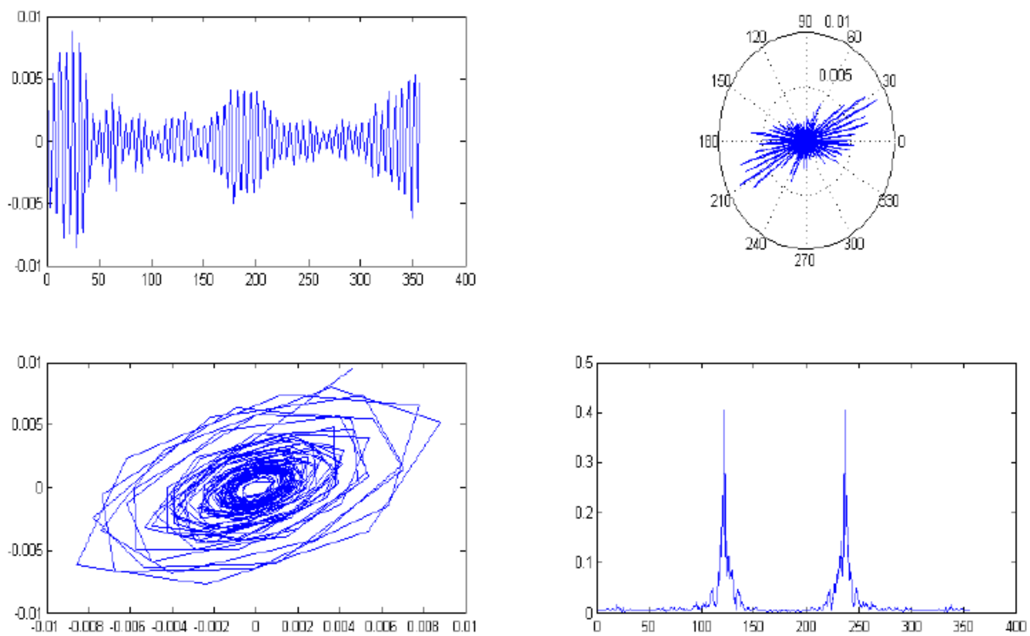

Figure 4 Waveform, Polar Plot, Phase Portrait and Spectrum of Ti/TiO2 sample, TiO_2 facing Transmitter

As seen from the plots, the spectrum of oxide side facing case shows wider bandwidths with more harmonic content than the metal facing case, as would be expected in X-Ray Diffraction studies, owing to the amorphous nature of the oxide causing irregular lattice and hence scattering [3]. Consequently, the phase portrait of the oxide facing case is much richer and ornamental, unlike clear periodic orbits seen in the metal facing case.

This observation of more chaoticity in the oxide facing case, due to the amorphous nature is further quantitatively confirmed by the observed high LLE value of 13.058, compared with the negative, non-chaotic LLE of -0.8231 observed for the metal facing case.

Thus, the degree of chaoticity in the scattered data as well as the spectral bandwidth serves as measures of amorphous nature in the material, and to validate this claim, nonlinear analysis is performed for two more samples, a 3cmx3cm 95% pure Aluminium sheet, yielding an LLE of -0.7565 and a thin-film sample of Zinc Oxide (ZnO) on Glass substrate, yielding a high LLE value of 10.3726, confirming the amorphous nature of ZnO.

3. Conclusion

The present work purports to the proposal, design and implementation of a microwave based material characterization technique, which essentially involves a transmitter-receiver antenna pair separated by a turn-table containing the sample to be characterized. By rotating the turn table 360 degrees, the distribution of intensity scattered from a microwave source is recorded, and nonlinear analysis techniques involving spectrum, phase portraits, polar plots and Lyapunov Exponents are applied to detect the crystalline/amorphous nature of the materials. The techniques are validated for nanostructured samples of Titanium Oxide as well as Zinc Oxide based thin films. It is hoped that these preliminary results will lay the foundation steps towards microwave based material characterization, with the significant advantage of lesser health hazard compared with X-Ray based techniques.

References

[1] W. Fahrner, Nanoprocessing and Nanoelectronics. Springer, 2003.
[2] R. Waser, Nanoelectronics and Information Technology, Advanced materials and novel devices, Wiley, 2003.
[3] R. Colclaser, S. Diehl-Nagle, Materials and Devices for Electrical Enginers and Physicists, Mc-Graw Hill, 1985.
[4] L. Solymar, D. Walsh, Lectures of the Electrical Properties of Materials, Oxford, 1979.
[5] Kennedy. G, and Davis. R, Electronic Communication Systems. Tata McGraw Hill, 2003.
[6] Philips, Charles L., John M. Parr, and E. Riskin. Signals, systems, and transforms. Prentice Hall, 1995.
[7] Gilmore, Robert, and Marc Lefranc. The topology of chaos: Alice in stretch and squeezeland. John Wiley & Sons, 2012.
[8] James, R. G., Burke, K., and Crutchfield, J. P. (2014). Chaos forgets and remembers: Measuring information creation, destruction, and storage. Physics Letters A, 378(30), 2124-2127.
[9] Rosenstein, M. T., Collins, J. J., and De Luca, C. J. (1993). A practical method for calculating largest Lyapunov exponents from small data sets. Physica D: Nonlinear Phenomena, 65(1), 117-134.