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Nondestructive Evaluation of Rubber Composites Using Terahertz Time Domain Spectroscopy

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Abstract

In recent years, with the progress of science, more and more detection methods are being used in various fields. However, the nondestructive testing of nonmetallic materials still needs further study. In this paper, an analysis of the time domain characteristics of rubber materials using terahertz detection technology was carried out, obtaining different defect rubber material spectral characteristics as well as imaging results and data. The results show that the THz spectrum imaging technique can detect the thickness of rubber material in the 0.1 ~ 4.0 terahertz band, and the image is clear and the resolution high. Meanwhile the time domain waveform obtained is sensitive to the debonding defects of the sample rubber, is suitable for judging the overall performance of the rubber inner defect detection, and can provide the scientific basis for rubber material performance.

Key words: *terahertz imaging, rubber composites, nondestructive testing, defects.*

Introduction

Terahertz (THz) radiation can be defined as the region between 0.1 terahertz and 10 terahertz, or the region of the electromagnetic spectrum between 3 mm and 30 μm in wavelength [1]. The terahertz "gap" is between the microwave region and infrared/optical region. As shown in *Figure 1*, the Terahertz wave is between the microwave and infrared, that is, the terahertz wave is an integration of micro-electronic , laser, and optical technology. The special nature of the terahertz wave contributes to the development of terahertz devices and the progress of terahertz research.

Terahertz (THz) time-domain spectroscopy (TDS) technology has developed rapidly over the past several decades and has been widely used in material identification, biomedicine and safety monitoring [2-4]. It has also been applied in nondestructive testing [5] and was evaluated as a non-destructive evaluation technique for the detection of composites. THZ time-domain spectroscopy is the reflection or transmission of rubber composites by terahertz waves interacting with them and then analyzing the effects of scattering, absorption, and the refractive index on each other [6].

Rubber is a highly elastic polymer material with reversible deformation. Rubber at room temperature is elastic, can produce great deformation under a small external force, and restore the status quo after the removal of external forces. Rubber is a completely amorphous polymer, its glass transition temperature (Tg) low, and the molecular weight is often large, more than a few hundred thousand [7]. The most widely used rubbers on the market are NBR, neoprene, EPDM, etc. These three kinds of rubber are widely used in medicine and health, commodity storage, electrical communication, civil construction and other aspects [8]. At present, many methods of testing rubber materials are damaging, with only the ultrasonic detection method [9] being non-destructive. However, the ultrasonic absorption of rubber material is too large, and it can only detect surface or sub-surface defects, while the detection of internal defects of rubber is incomplete. In this paper, terahertz time-domain spectroscopy was applied to the non-destructive testing of rubber materials, which could realise thickness detection and debonding detection, and provide a strong basis for judging rubber material defects.

Extraction of optical parameters of rubber materials

Theoretical method

In this experiment, the transmission method was used to extract the material parameters. *Figure 2* shows the detection schematic diagram.

After the incidence of the TeraHertz wave $E_{THZ}(\omega)$ on the dielectric surface, $E_{t0}(\omega)$ represents the signal directly transmitted to the receiving end, $E_{tm}(\omega)$ the signal to the receiving end after several reflections, and $E_{ref}(\omega)$ represents the reference signal obtained when there are
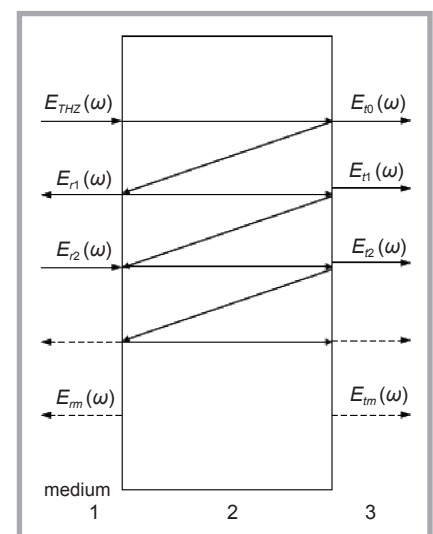

Figure 1. Terahertz spectrum diagram.

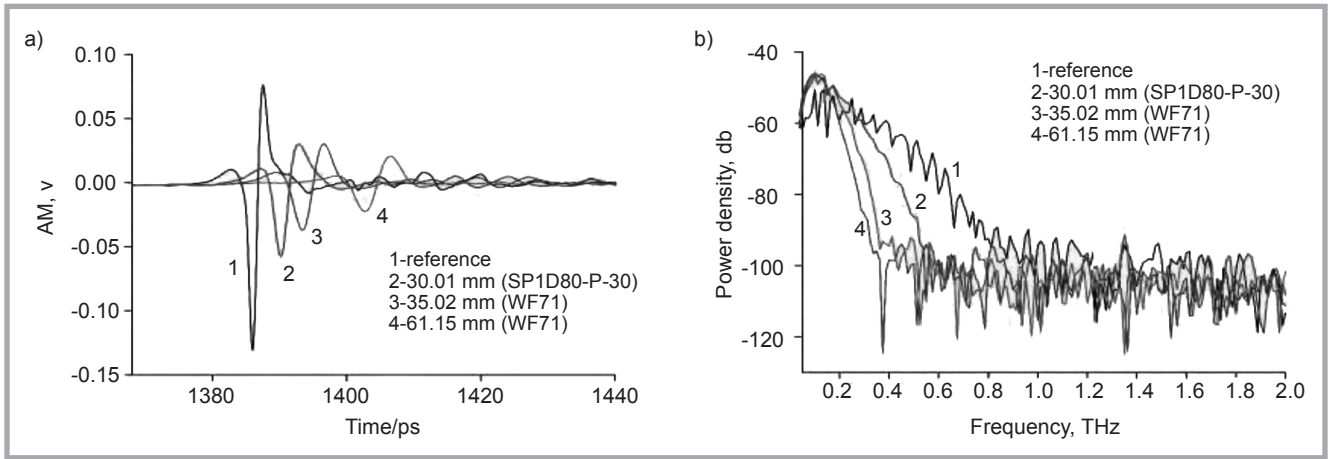*Figure 2. Terahertz – wave incident and reflection paths.*

Figure 3. Time-domain and frequency-domain spectra of chloroprene rubber with different thicknesses.
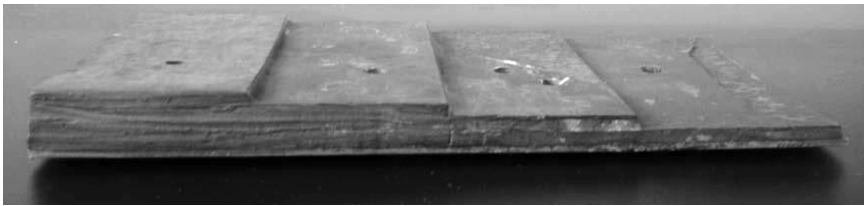

Figure 4. Stepped rubber bonded specimen.

no samples. $E_{t1}(\omega)$ and $E_{t2}(\omega)$ refer to the first and second reflection signals of the terahertz wave. The ratio of $E_{ref}(\omega)$ and $E_{sam}(\omega)$ ($E_{sam}(\omega)$ refers to the sum of the transmission signals when there are samples) represents the complex transmission function *Equations (1)* and *(2)*.

In the *Equations (1)* and *(2)*, $FP(\omega)$ represents the change factor caused by repeated reflections, and m the mth transmitted wave. Since the thickness of the samples are large and the distance between the first and second back waves long, $FP(\omega)$ approximates to 1.

If the thickness of the samples is small, $FP(\omega)$ must be obtained through *Equation (2)*. The complex transfer functions are expressed in the form of the modulo and radii: $H(\omega) = \rho(\omega)\exp[-j\varphi(\omega)]$ and the complex refractive index $\tilde{n}(\omega) = n(\omega) - j\kappa(\omega)$ are substituted into *Equation (1)*, then the following equations can be obtained *Equations (3)* and *(4)*.

The complex refractive index $\tilde{n}(\omega)$ and permittivity $\varepsilon(\omega) = \varepsilon_r(\omega) - j\varepsilon_i(\omega) = (n - j\kappa)^2$ can be obtained by the iterative method. n refers to the refractive index, κ to the extinction coefficient, ω to the frequency,

d to the thickness of the samples, $\rho(\omega)$ to the amplitude of $H(\omega)$, and $\varphi(\omega)$ refers to the phase postion of $H(\omega)$. Optical parameters such as the refractive index $n(\omega)$, extinction coefficient $\kappa(\omega)$ and absorption coefficient $\alpha(\omega)$ can be obtained by mathematical derivation as follows:

$$n(\omega) = \varphi(\omega)\frac{c}{\omega d} + 1 \qquad (5)$$

$$\kappa(\omega) = In[\frac{4n(\omega)}{\rho(\omega)(n(\omega)+1)^2}]\frac{c}{\omega d} \qquad (6)$$

$$\alpha(\omega) = \frac{2\omega}{c}\kappa(\omega) = \frac{2}{d}In[\frac{4n(\omega)}{\rho(\omega)(n(\omega)+1)^2} \qquad (7)$$

Extraction of optical parameters of the sample rubber

Effect of sample rubber thickness on optical parameters

In this experiment, we took a certain particular rubber as an example and chose different thicknesses of the rubber to perform experiments in order to obtain the spectral properties of the same kind of material with corresponding thicknesses, as shown in *Figure 3*.

Figure 3 shows the time-domain spectra of chloroprene rubber with different thicknesses (1 is the reference signal, which is the time-domain signal of the terahertz wave through atmospheric sampling, 2 a sample piece with a thickness of 30.01 mm, 3 with 35.02 mm, and 4 with 61.15 mm). From the time-domain signal, it can be observed that the flight time of 4 is longer than that of 2 because the thickness of 4 is thicker from the viewpoint of the flight time. From the amplitude, that of 4 is also significantly smaller than for 2, which can also be reflected in the Terahertz spectrum after the

$$H(\omega) = \frac{E_{sam}}{E_{ref}} = \frac{4\tilde{n}}{(1+\tilde{n})^2}[\exp\{\frac{-jd[\tilde{n}(\omega)-1]\omega}{c}\}]FP(\omega) \qquad (1)$$

$$FP(\omega) = \frac{1-(\frac{1+\tilde{n}}{1-\tilde{n}})^{2m+2}\exp[-j(2m+2)\tilde{n}(\omega)d\omega]}{1-(\frac{1+\tilde{n}}{1-\tilde{n}})^2\exp[-j2\tilde{n}(\omega)d\omega]} \qquad (2)$$

$$\rho(\omega) = \frac{4[n^2(\omega)+\kappa^2(\omega)]^{1/2}}{[n^2(\omega)+1]^2+\kappa^2(\omega)}\exp[-\frac{\kappa(\omega)d\omega}{c}] \qquad (3)$$

$$\varphi(\omega) = \frac{[n(\omega)-1]\omega d}{c} + \arctan[\frac{\kappa(\omega)}{n^2(\omega)+n(\omega)+\kappa^2(\omega)}] \qquad (4)$$

Equations (1), (2), (3) and (4).

Fourier transform. Transforming from the Fourier to the frequency signal, we can see that different thicknesses are all absorbed to a various extent. However, the thicker the material, the more serious the absorption, and the stronger the scattering phenomenon, thus we can know both the transmission and reflection power spectrum attenuation are more serious above 0.6THZ. In the experiment, the thickness of the rubber sample has an important influence on the acquisition of optical parameters. If the sample is too thin or too thick, the measurement accuracy can be affected. This is because the thickness of the rubber sample will cause the terahertz spectroscopy system to generate a terahertz wave to interact with many molecules [10]. If the rubber sample thickness is too thick, it can reduce the signal to noise ratio to a certain extent because of the role of pulse absorption. While a too thin rubber sample will increase the difficulty of distinguishing between the reference signal and rubber sample signal, which is related to the absorption of the rubber material itself.

Detection of the adhesive thickness of rubber material using terahertz time-domain spectroscopy

Rubber material defects are as follows: uneven thickness, delamination, degumming, inclusions and aging. For the uneven thickness problem, the existing means of detection is to measure from the metal surface with an electromagnetic force and the measurement results are not satisfactory. The best scheme for detecting the rubber thickness is to measure from the rubber aspect. As long as the optical parameters are extracted and the refractive index is accurate, measuring the thickness of rubber with terahertz will be very accurate [11]. Moreover if it is multi-layer rubber bonding, you must first know the optical parameters of each layer of rubber to carry out a combined analysis. For stratification and degumming detection, the previous application is ultrasonic measurement. But the ultrasonic attenuation in the rubber material is relatively large, and hence the detection accuracy is not high. For the detection of aging, this was previously realised through an aging test. However, the defects of some rubber seals are not seen before aging, only to be seen afterwards. Therefore to solve this problem, aging can now be detected by terahertz, so that the degree of aging can be detected in advance to facilitate analysis of the service life. Generally the defect detection of

rubber materials is commonly seen in the sealed cabins of aircraft and ships as well as in high temperature and high pressure water and oil seal rings, etc.

Sample rubber and parameter settings
Specimen name
Specimen size
The specimen size was about 265 * 123 * 20 cm in this experiment (**Figure 4**).

Detection step distance
The detection step distance was 0.5 mm in this experiment.

Detection process and analysis
The test piece was a ladder-type rubber bonded test specimen, which was placed on a terahertz time-domain spectrum detection device platform (**Figure 5**). Using reflection imaging and flight time imaging algorithm simulation, both thickness detection [12] and delamination testing were able to be carried out on the specimen. (1) rubber material thickness detection using terahertz time-domain spectroscopy.

Observing the material properties, part of the material surface was damaged, presenting a trap cut shape. Seen from a lateral
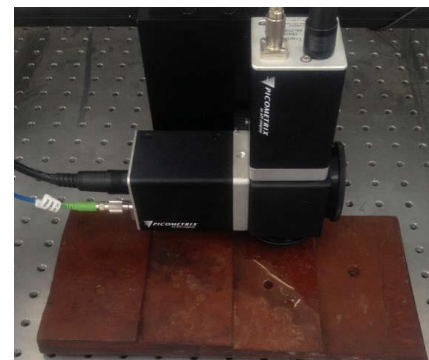

Figure 5. *Terahertz time – domain spectroscopy detector.*

view, the material was divided into multiple layers of rubber, as shown in **Figure 4**.

Through the gray scale map and B-Scan diagram, the thickness change can be visualised.

As shown in **Figure 6**, although the image is a gray-scale imaging map, its color is divided into five levels according to the depth of color. The surface of the material is divided into four step layers, with the first area relatively thin due to the first step layer's surface damage. Through
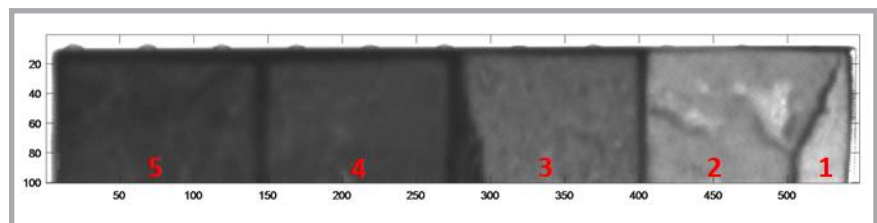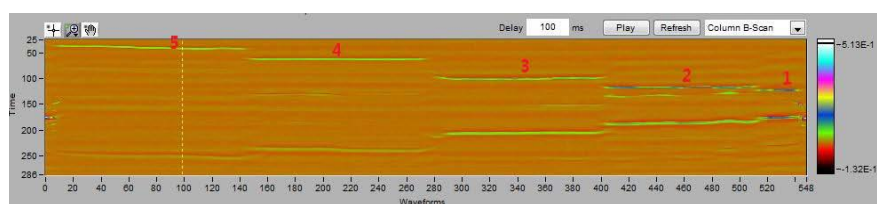

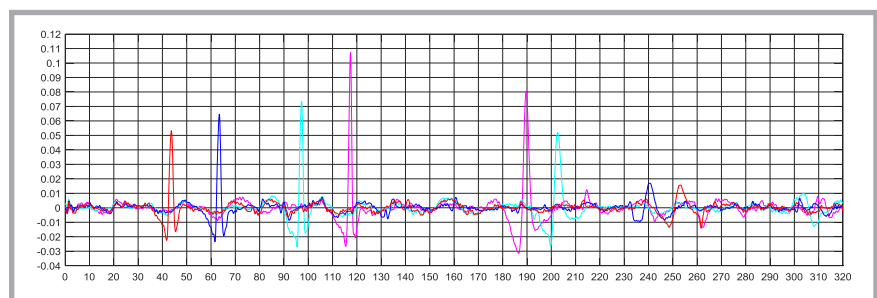**Figure 6.** *Gray-scale images of test results.*

Figure 7. *B-Scan test results*

Figure 8. *Detection of the waveform.*

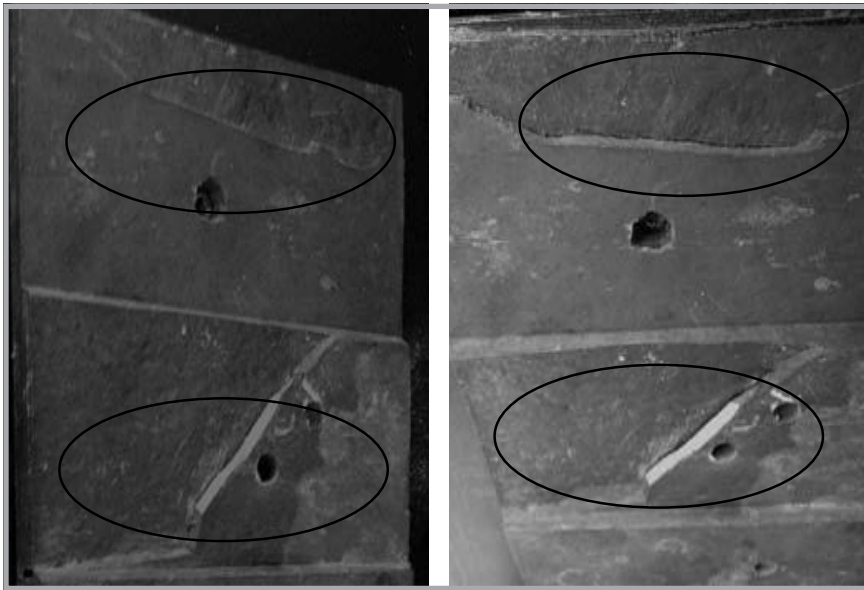**Figure 9.** *Local layered material map of detection material.*

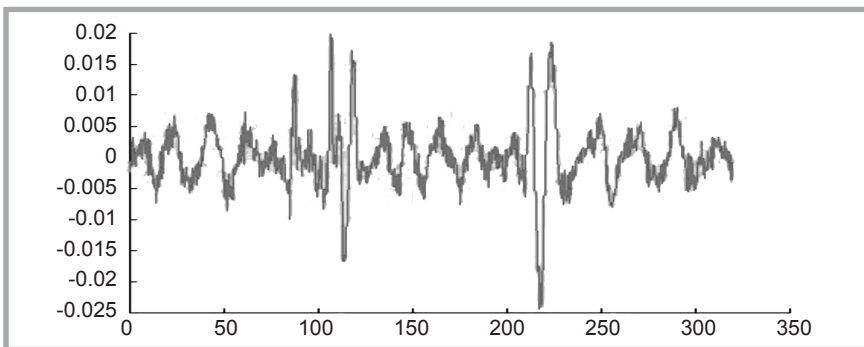**Figure 12.** *Time domain waveform of feature point 1.*

ence of the time domain. The red waveform is the first trapezoid, the blue waveform – the second trapezoid, the cyan – the third trapezoid, and the pink is the fourth trapezoid, as shown in *Figure 4*. As the thickness of the rubber increases, the flight time increases and the energy decreases. The upper and lower layer time delays are as follows: the first ladder layer 251.5-40.7 = 210.8 ps, the second ladder layer 240.7-63 = 177.7 ps, the third ladder layer 206.6-101.9 = 104.7 ps, and the fourth ladder layer 188.7-117.4 = 71.3 ps.

The thickness of different regions can be calculated through the refractive index (1.2214) and absorption coefficient: red ladder thickness 16.2 mm, blue ladder 13.6 mm, blue ladder 8 mm, and pink ladder 5.4 mm. It is also possible to make a thickness measurement by making a reference sample and using the result as a data reference.

Rubber material degumming and delamination detection using terahertz time domain spectroscopy

Material stratification detection [13] is carried out. *Figure 9* shows the picture of a real material product with obvious surface layer defects. *Figure 10* shows the grey scale imaging of local area deection.

For the detection result image above, its B-Scan image was analysed. The horizontal direction is the Y-axis, and the longitudinal direction is the X-axis. By enlarging the B-Scan map to compare its corresponding position with the physical map, we can see that the adhesive layer at the first gradient is obviously layered, and after the measurement delamination appears at a distance of 3 mm above the surface layer. A third gradient also appears in a hierarchical state, which can be specifically analysed by comparing the corresponding waveform. The B-Scan diagram shows the material stratification
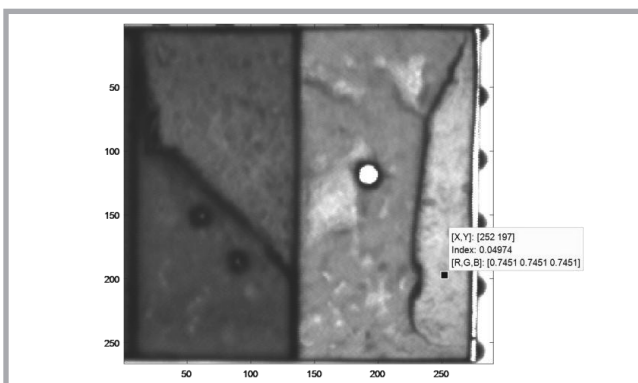
analysis of the regional colour, a qualitative analysis of the different thickness of the material can be carried out.

From *Figure 7*, we can see that the flight time in terahertz of the material presents a ladder-type change, in accordance with *Figure 6*. Hence the thickness changes of each region can be analysed. Similarly according to the flight time graph, the

thickness of the sample can be calculated through refractive index calculation. *Figure 8* shows the time domain waveforms of the four regions. The waveforms are matched against the thicknesses so as to analyse the time difference to calculate the thickness.

In *Figure 8*, the thickness is calculated through analysing the flight time differ-

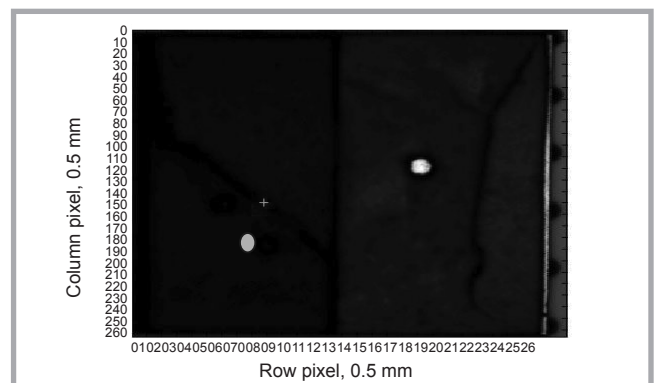**Figure 10.** *Grey scale imaging of local area detection.*

Figure 11. *Grey scale map of feature point 1.*

area. The abnormal area in the figure can be determined as the hierarchical area of the rubber material.

To perform layer detection, we can use the time-domain waveform and B-Scan plot to analyse the specific delamination of different sections, selecting different section points on the grey-scale map. *Figures 11-14* show the grayscale image, time-domain waveform image, row B-Scan map and column B-Scan map of the feature point location selected.

As shown in *Figure 15*, the selection of feature points are two hole edges. It can be seen from the time-domain waveform (*Figure 16*) that the two hole edges are severely affected by the edge effect with sharp attenuation (the former wave crest should be at about 98 ps) and the inside stratification is serious. *Figure 17* shows the raw B-Scan map of feature points 2 and 3. Through B-Scan diagram analysis, we can observe that the green point is in the 140 mm position. The column B-Scan (*Figure 18*) shows that the 140 m position is a hole and that the layering is severe.

Figure 19 shows the grey-scale map of feature point 4. In the grey-scale map, feature point 4 is in the 160 mm position. From the time domain waveform diagram (*Figure 20*), it can be seen that the layer has three peaks and that a layer exists. Then through the B-Scan diagram (*Figures 21* and *22*) we can ascertain that the stratification layer size is of 21 ps and 2.5 mm through refractive index calculation.

■ Conslusions

In summary, terahertz spectroscopy is a non-contact measurement technology, with the advantages traditional detection methods do not have. Terahertz spectrum detection technology can use T Hz-TDS technology to perform effective identification and determination on a rubber structure with respect to its morphology and environmental impact.

■ Nondestructive testing can be carried out using terahertz time-domain nondestructive testing technology. The thickness of the material can be analysed by three methods: image detection, B-Scan and detection waveforms; and the refractive index and absorption rate can be used to accurately calculate the thickness of a rubber material.

■ Terahertz time-domain spectroscopy can be used to carry out layer detection inside a rubber material. By analysing the detection gray-scale, time-domain
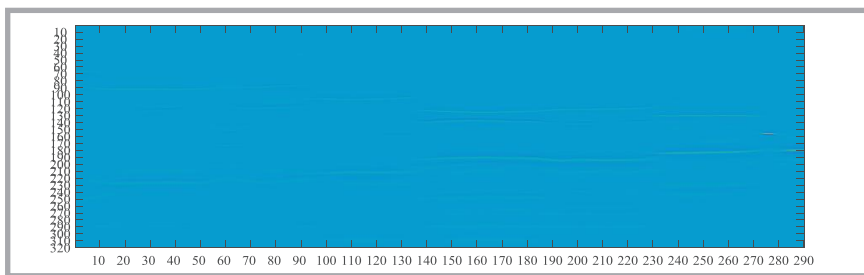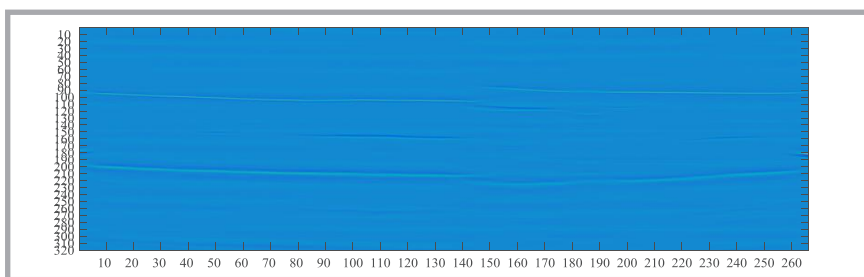

Figure 13. Row B-Scan map of feature point 1.

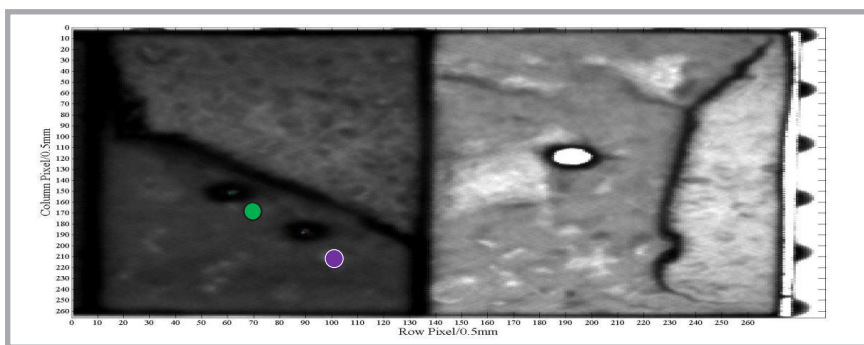*Figure 14. Column B-Scan map of feature point 1.*

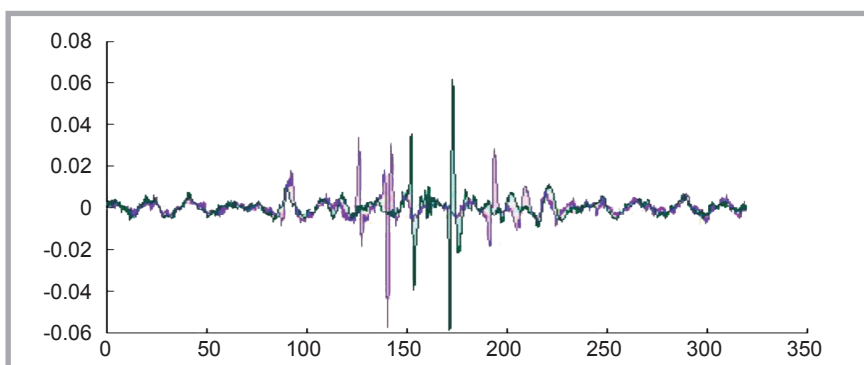*Figure 15. Grey-scale map of feature points 2 and 3.*

Figure 16. Time-domain waveform comparison of feature points 2 and 3.
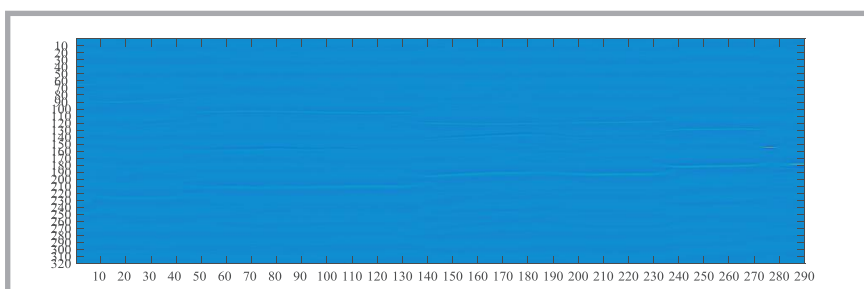

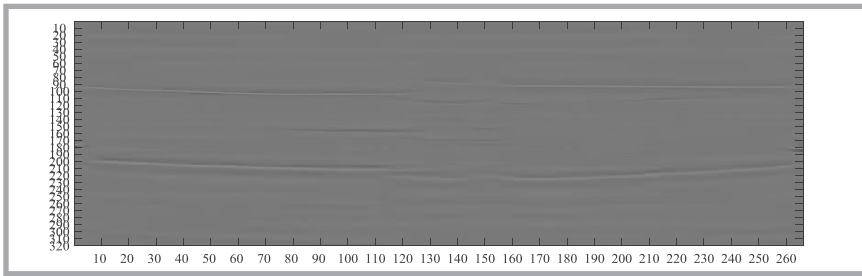*Figure 17. Row B-Scan map of feature points 2 and 3.*

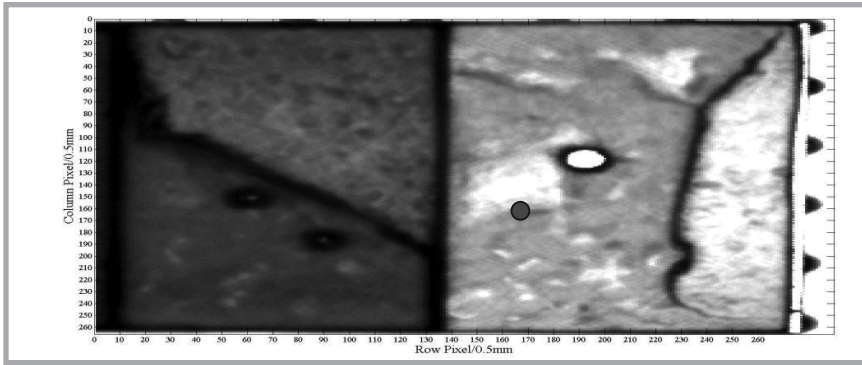*Figure 18. Column B-Scan map of feature points 2 and 3.*
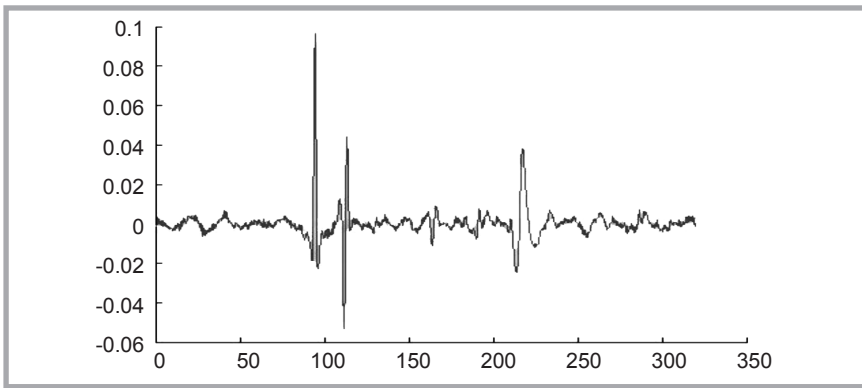

Figure 19. Grey-scale map of feature point 4.

Figure 20. Time domain waveform of feature point 4.
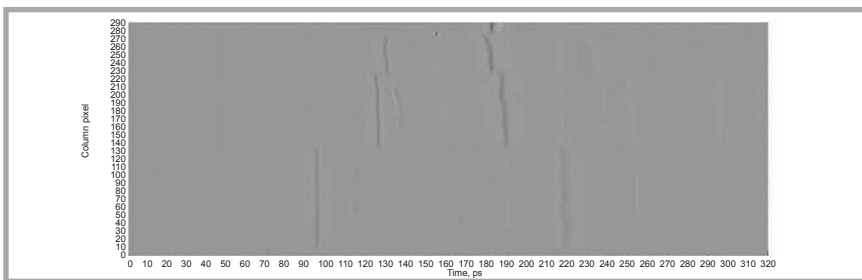

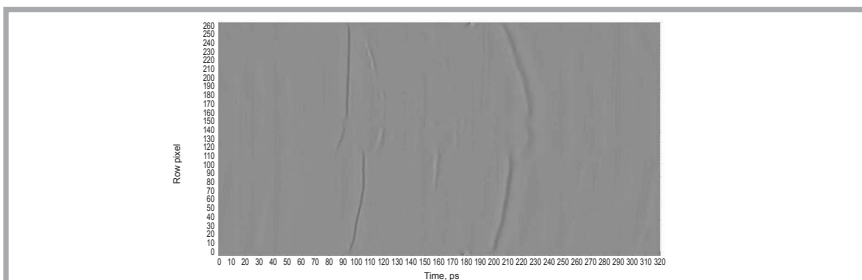*Figure 21. Line B-Scan of feature point 4.*

Figure 22. Column B-Scan of feature point 4.

waveform and B-Scan map, the stratification position can be qualitatively determined. Besides this, calculation of the refractive index can accurately determine the size of the layer.

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