

Terahertz Spectroscopic Analysis and Multispectral Imaging of Epitaxially Grown Semiconductors with Nanometer Resolution

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Abstract

Terahertz spectral analysis has been conducted on epitaxially grown semiconductor structures. Epitaxially grown semiconductors are important for microelectronic and optoelectronic devices and also for integrated circuits fabricated using semiconductors. In this paper, we report results of terahertz time-domain spectroscopy of grown SiGe layers on Ge buffer and separately a Ge buffer that was grown on a Si <001> wafer. In particular, evolution of the time-domain spectra as a function of thickness of both samples was investigated by the terahertz pump-probe technique. Representative spectra were analyzed to determine the respective layers' spectral signatures. It was found that the spectroscopic analysis uniquely identified different layers by characteristic absorbance peaks. In addition, terahertz imaging was conducted in a non-destructive, non-contact mode for detecting lattice stacking fault and dislocations. Sub-surface imaging of grown SiGe layers on Ge buffer and that of the Ge buffer grown on a Si wafer reveals interesting lattice features in both samples. A comparison with TEM images of the samples exhibits that the terahertz image reproduces the dimensions found from TEM images within the experimental error limits. In particular, 3D images of both samples were generated by the terahertz reconstructive technique. The images were analyzed by graphical means to determine the respective layer thicknesses. Thus, this technique offers a versatile tool for both semiconductor research and in-line inspections.

Keywords: Terahertz; Reconstructive multispectral imaging; Sub-surface analysis; Non-destructive; Non-contact; Layer by layer inspection; Terahertz time-domain spectroscopy; Sub-surface spectral analysis; Nanometer resolution

Introduction

For many integrated circuit and high speed transistor applications the strain in the epitaxial layers of a semiconductor is relieved by introducing misfit dislocations that has inherent strain in their lattice structure. As indicated in reference [1], a fully relaxed Si_{0.7}Ge_{0.3} layer is required as the buffer layer for high mobility field effect transistors having strained Si or strained SiGe channels. Therefore, controlling the growth conditions and a strategic design of the structure is necessary for minimizing the density of dislocations threading through device layers grown on top of the relaxed buffer layer. While visualization of these structures on nanometer scale is important [2], a time-domain spectroscopic investigation will yield additional information about the molecular nature of these structures that are also important. Especially, terahertz spectroscopic inspection on a layer-by-layer basis could play a key role for the success in creating controlled 3D semiconductor structures. The need to prevent strain relaxation in thin layers or to control the density and distribution of defects in relaxed structures has led to extensive research on strain relaxation mechanisms and also on the properties of the defects which are required to relieve the strain. Therefore, in addition to the spectroscopic analysis, 3D visualization of these structures on a nanometer scale is of paramount importance. In particular, the capability of sub-surface inspection on a layer-by-layer basis in a non-destructive and non-contact mode is the key for success in creating such 3D structures. Effective testing for minimized wafer rejection during process development and beyond was discussed elsewhere [3].

In what follows, we describe terahertz time-domain spectroscopy and high resolution reconstructive imaging via terahertz reflectometry for strain relaxed SiGe/Ge/Si structures. Two cases are investigated. Grown Ge layer on Si <001> substrate is inspected by terahertz pump-probe signals as a function of thickness yielding layer-by-layer spectroscopic information of the epitaxial structures. Then, another

sample is inspected where SiGe layer was grown on top of the Ge buffer of the first case, also via layer-by-layer terahertz pump-probe technique. The spectra are analyzed for their identifying features of the epitaxially grown semiconductor layers. In addition, both samples were inspected by 3D sub-surface imaging. A side by side comparison is attempted. Clear visualization of the lattice stacking fault and defects was demonstrated that could shed light for streamlining the process parameters. We utilized terahertz non-contact multispectral reconstructive imaging [2] to investigate aforementioned grown semiconductor layers. An algorithm of gridding with inverse distance to power equations has been used for reconstructive imaging. The images are further analyzed via graphical means to measure the interface features and layer thicknesses.

Time-Domain Spectrometry

Terahertz time-domain spectroscopy has attracted attention from a broader scientific community in the recent years for its potential to probe delicate materials without imparting ionizing perturbation. Details of terahertz generation via dendrimer dipole excitation (DDE) have been described elsewhere [4]. We utilize the property that the terahertz radiation (T-ray) can be focused on a given layer below the surface, thus allowing layer-by-layer investigation capability. The lowest layer thickness for probing is defined by the scanner step size. The setup used for the present investigation has a resolution of ~24 nm.

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The experimental arrangement is shown in Figure 1. Here, a compact design of an integrated nanosensing terahertz spectrometer system with fiber-coupled beam delivery has been realized by arranging the components in the optimized fashion as shown. All instruments are from Applied Research & Photonics (Harrisburg, PA 17111, USA) (Figure 1).

The fixed mirrors in Figure 1 helps adjusting the path length of the probing beam. It was determined that a fixed ratio of 10:1 intensities for the pump to probe produces the sharpest time-domain interference, usually known as the interferogram. An interferogram is generated when the stationary beam is scanned by the terahertz beam whose intensity distribution is captured by the detector pair for both the reflected and the transmitted intensity over a few pico-seconds. Special care was taken in the design so that the pump to probe intensity ratio remains fixed throughout the operation. All operations are made at room temperature for different kinds of samples such as solids, liquids and gaseous.

When the T-ray interacts with molecules of a sample, it may stimulate many resonances such as molecular vibrations, rotation, translation, torsion, and/or other resonances in the system (in general molecular “events”), resulting in the T-ray photons being affected by characteristic amounts determined by a specific interaction or event. The change in energy and/or frequency yields information about the molecular nature of the interaction. Infrared and Raman spectroscopy yields similar information but not capable of detecting as many resonances as can be detected with T-ray because terahertz photons are sensitive to the motions of the entire molecule as opposed to just a bond or charge state [5]. Molecular simulation reveals that there are many resonance states that exist when a molecule faces ambient perturbation [5]. As most material remains at its lowest energy state under normal and steady state conditions, T-ray will probe and yield corresponding states in the absorbance spectrum.

Terahertz imaging with nanometer resolution

Reconstructive imaging is described in details elsewhere [2]. In a nutshell, reconstructive imaging is used as an alternative for CCD based imaging such as in digital microscopes and cameras. CCDs have limitations for achieving higher resolution, because, the pixel size is defined by the size of the array elements of the sensor/CCD used for image generation. Also, it is strictly a surface imaging device. TEM offers high resolution imaging; however, it is strictly a destructive

technique with laborious sample preparation requirements and only for small geometries. Other techniques such as focused ion beam, XRD, etc., are also destructive technique. Reconstructive imaging offers an important opportunity to define the pixel size (or voxel size) by a hardware and software combination. Here the response from an object is recorded on a three dimensional space as it is rasterized. The recorded (scanned) response is then converted to 3D image via an algorithm. As depicted in Figure 2, a simple function, $f(x,y,z)=c*\cos(x)$, was used to test the validity of the computation over a 3D space (Figure 2).

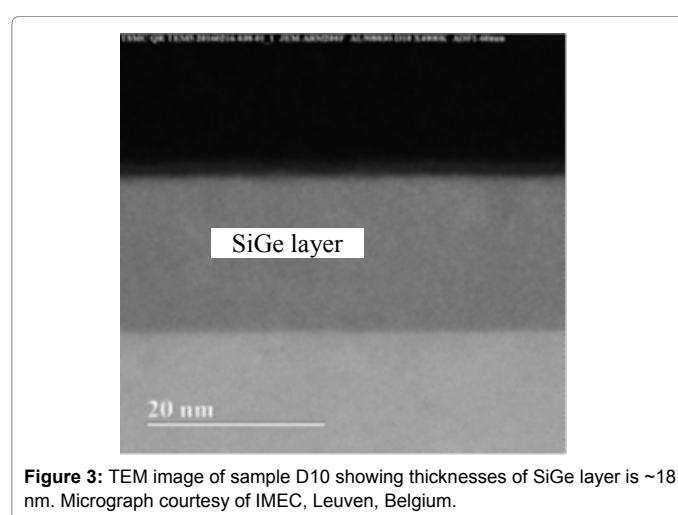
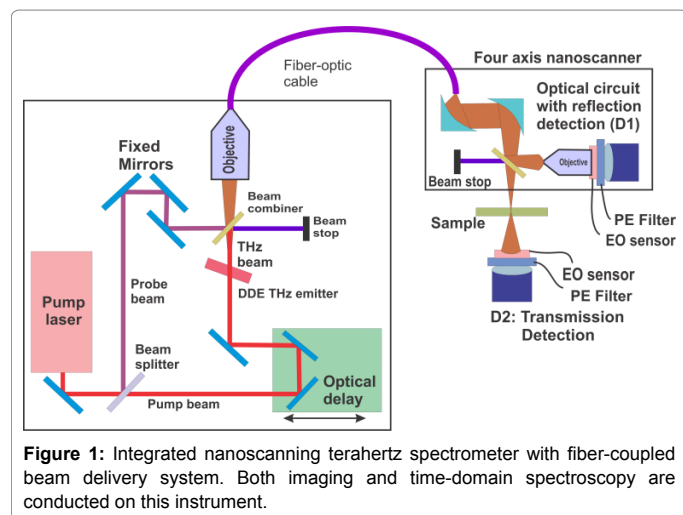
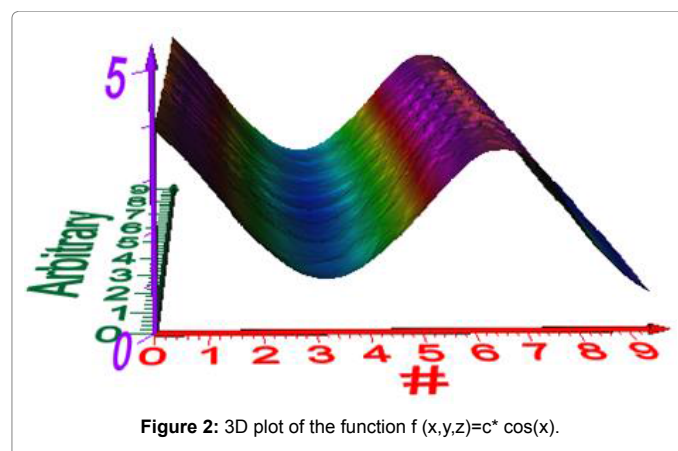
Experimental Results and Discussion

Here we investigate the following semiconductor samples. The first sample is a Ge buffer grown on Si<001> substrate (termed: D02). The Ge buffer is expected to be ~600 nm thick per data provided by the supplier (via personal correspondence with the IMEC, Leuven, Belgium). The second (termed: D10) contains a SiGe top epitaxial layer grown on Ge buffer on Si <001>, where the SiGe is reported to be 18 nm as determined by TEM (Figure 3).

Described below are the data from terahertz time-domain and frequency domain spectroscopic observations of both samples indicated above. After that image analyses of both samples are presented.

Terahertz spectroscopy

Figure 4 shows the evolution of terahertz time-domain signal of Sample D02 as a function of thickness, t . Total 1225 nm depth was



scanned from top to downwards at a step length of 25 nm. Selected signals from Figure 4 were analyzed via Fourier transform (Figure 5); one at $t=0$ nm (surface) and the other one at $t=600$ nm depth, which is on the Si substrate.

Figure 5 exhibits the absorbance spectra for Ge ($t=0$ nm) and for Si ($t=600$ nm) over 0.1 THz to 10 THz range, obtained by Fourier transform of two respective time-domain signals from Figure 4. Low frequency absorbance peaks are visible at 0.6 THz and 1.3 THz due to ambient moisture. At frequencies above 2 THz, both Ge and Si exhibit their own absorbance peaks; some of which overlap but a majority of the absorbance peaks are separated. These peaks are due to each materials' own characteristic properties, thus represent a unique signature for the respective materials. For example, silicon spectrum (red curve) shows an absorbance peak at 3.41 THz. According to spectrum from THz database [6], silicon has a prominent absorbance peak at approximately 3.5 THz. The observed peak at 3.41 THz of the present work is a close match, thus indicating a signature for Si (Figures 6 and 7).

Figure 6 shows the thickness dependent evolution of terahertz time-domain signal of Sample D10 over thicknesses $t=0$ nm to $t=1225$ nm from top to downwards at a step length of 25 nm. These time-domain signals (from Figure 6) were analyzed via Fourier transform as above. Figure 7 exhibits the absorbance spectra for SiGe ($t=0$ nm) and for Ge ($t=25$ nm) over 0.1 THz to 10 THz range. Low frequency absorbance peak is visible at 0.6 THz due to ambient moisture. At frequencies above 2 THz, both SiGe and Ge exhibit their own characteristic peaks; some of which overlap but most of the absorbance peaks separated.

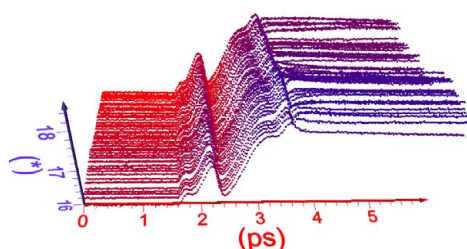


Figure 4: Thickness (t) dependent evolution of the terahertz time-domain signal of Sample D02. Total 1225 nm depth was scanned at a step size of 25 nm. *The vertical axis values are in counts \times 3000.

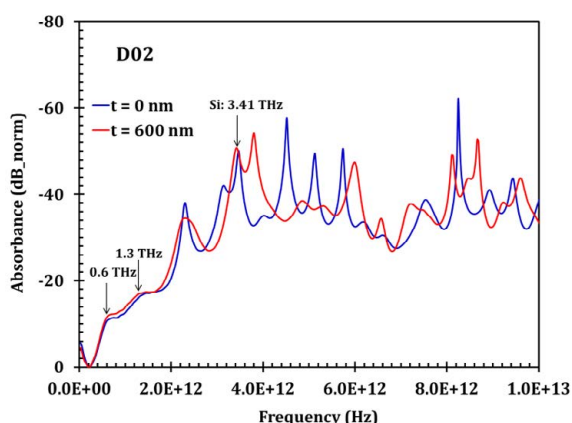


Figure 5: Absorbance spectra of Sample D02 at thickness ($t=0$ nm) (surface, Ge) and $t=600$ nm. Significant shifts in peaks occurred between Ge ($t=0$ nm) and Si $\langle 001 \rangle$ ($t=600$ nm). (Low frequency peaks at 0.6 THz and 1.3 THz are due to absorption by moisture at ambient conditions).

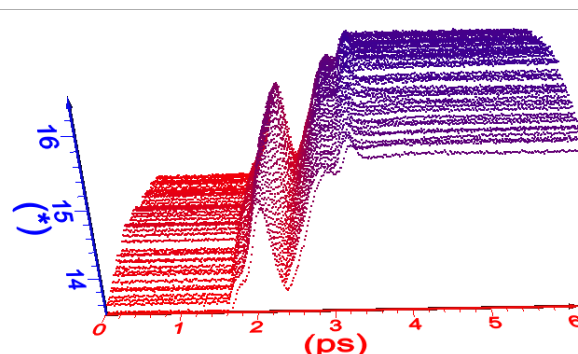


Figure 6: Evolution of terahertz time-domain signal of Sample D10 as a function of thickness. Total 1225 nm depth was scanned at a step size of 25 nm. *The vertical axis values are in counts \times 3000.

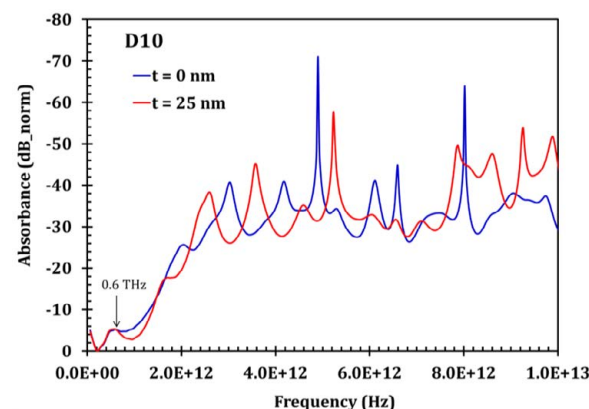


Figure 7: Absorbance spectra of Sample D10 at thickness $t=0$ nm (surface, SiGe) and $t=25$ nm (Ge). Significant shifts in peaks occurred between Ge ($t=0$ nm) and Si $\langle 001 \rangle$ ($t=600$ nm).

These peaks are due to each materials' own characteristic properties, thus represent a unique signature for the respective materials.

Figure 8 exhibits a comparison of two Ge spectra, one obtained at the surface of sample D02 and the other is under the SiGe layer of sample D10. The spectrum at the surface of sample D02 exhibits prominent moisture absorption peak but the spectrum of buried Ge under the SiGe layer at a depth of 25 nm shows significantly reduced moisture absorbance peak. Also, the absorbance peaks at the depth are shifted compared those at the surface, indicating the surface Ge is likely to be oxidized by the ambient oxygen. Under this assumption, this technique thus offers a way to discerning between pure and oxidized Ge and other elements (Figure 8).

Terahertz imaging

Figures 9-16 exhibit the terahertz imaging and analysis of these two samples mentioned above. Figure 9 shows one μm^3 volume of Ge grown on Si (sample D02). It is notable that this is the first time a 3D lattice image has been produced for these samples. Unlike, a TEM image which shows only a single surface; the images of present paper are true 3D and allows inspection of the specimen on a layer by layer basis. A single face (side surface) of the cube is shown in Figure 10. Figure 11 shows a graphical analysis of the lattice structure of Figure 10. The Ge buffer's thickness was determined to be ~ 590 nm which is in agreement with the TEM image of 600 nm (by personal correspondence with the IMEC labs, Leuven, Belgium).

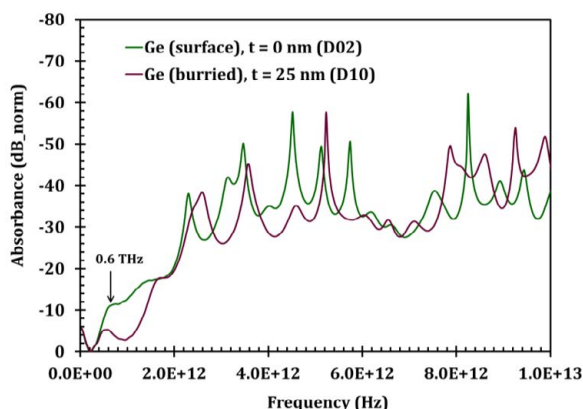


Figure 8: Comparison of Ge spectra both at the surface of sample D02 and under the SiGe layer of sample D10. The spectrum for the surface (D02) exhibit prominent moisture absorption peak.

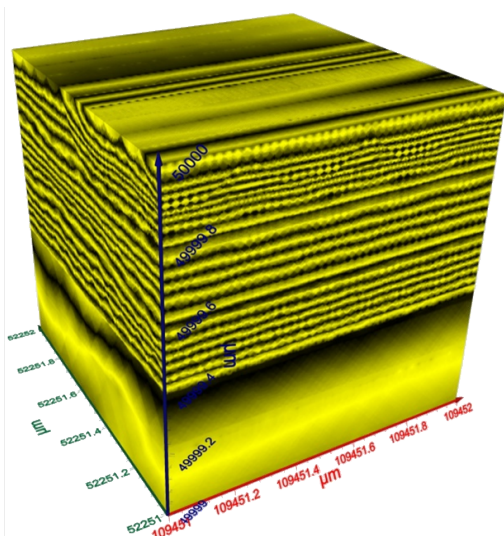


Figure 9: Sample D02: one cubic micron volume showing the Ge buffer's lattice structure grown on Si <100> substrate. Ge buffer's lattice planes are visible on top of the Si substrate.

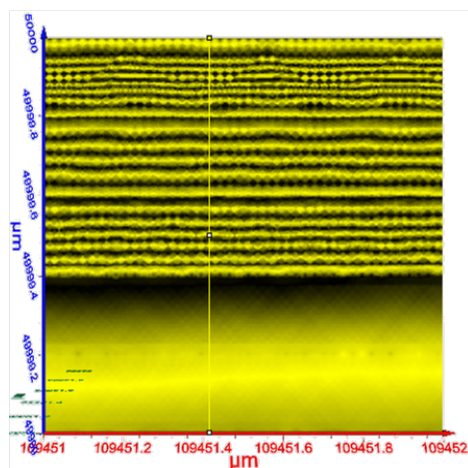


Figure 10: Image of a single side of sample D02 extracted from Figure 9. One square micron face (XZ surface) is used for graphical analysis.

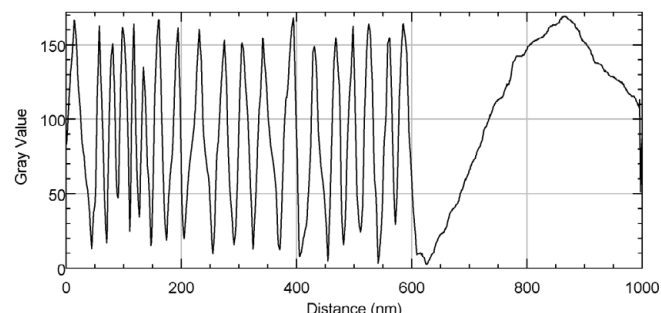


Figure 11: Graphical analysis along the vertical line of sample D02, see inset of Figure 10. Measured thickness of the Ge layer is ~590 nm (see arrow).

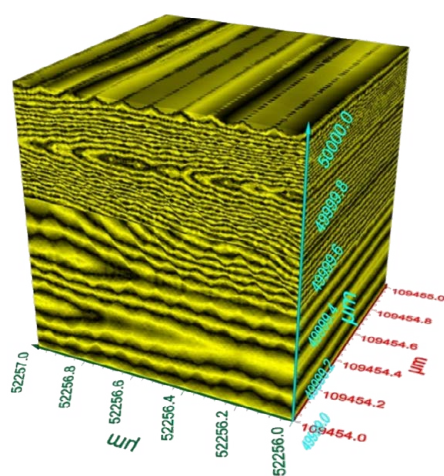


Figure 12: Sample D10: One cubic micron volume showing the lattice structure of Ge buffer and grown SiGe layer on the top. The top layer (SiGe) is ~18 nm. Ge buffer's lattice planes are visible below the SiGe layer followed by Si lattice structure at the lower part.

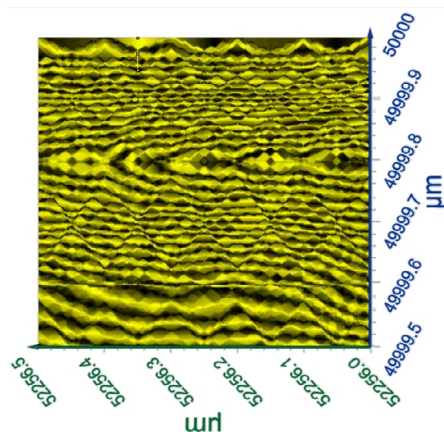


Figure 13: Image of 500 nm x 500 nm surface extracted from Figure 12 for sample D10. Graphical analysis of the top layer is shown in Figure 14.

Figure 12 shows a one μm^3 volume of sample D10 and Figure 13 shows a slice of 500 nm x 500 nm surfaces extracted from Figure 12. A graphical analysis of the top layer of the image in Figure 13 is shown in Figure 14. The thickness of the SiGe (top) layer is ~18 nm; this is the same thickness as determined by TEM image (Figure 3). Some features of the size of ~1 nm were also observed in the SiGe layer as shown in

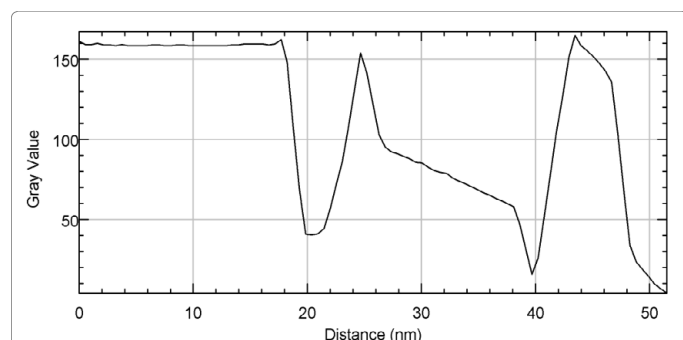


Figure 14: Graphical analysis of sample D10 from the image in Figure 13. Measured thickness of the SiGe layer (top) is ~18 nm (see arrow). Ge lattice structure is clearly visible.

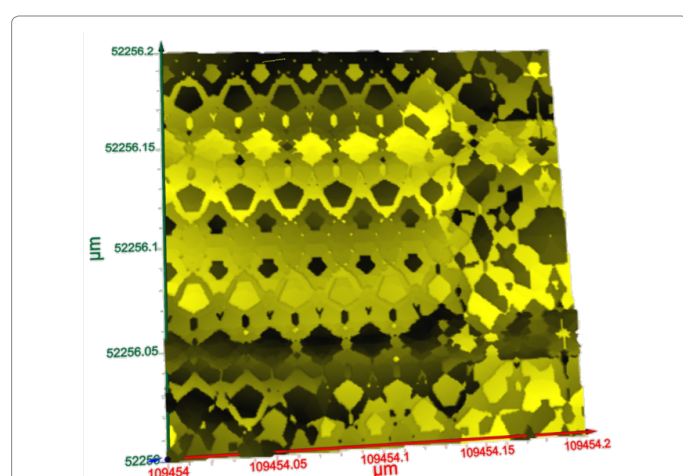


Figure 15: Nanometer size particulate features visible in the SiGe layer of sample D10. A single particle's (red arrow) size is shown in Figure 16. Other nano-particles are also visible with diameter less than or equal to a nanometer.

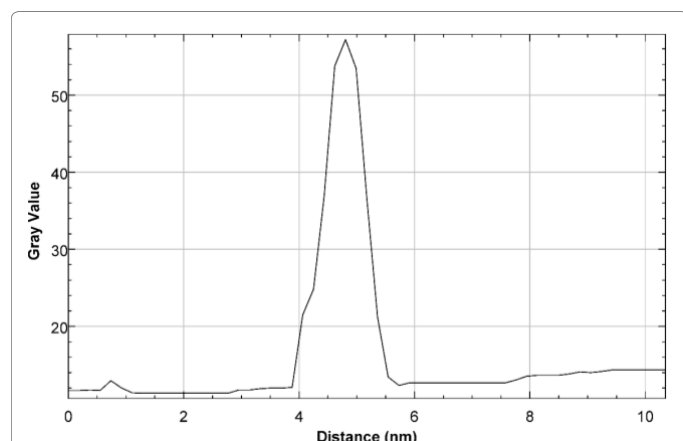


Figure 16: Graphical analysis of a nano-particle in the SiGe layer of sample D10. A single particle has a diameter <1 nm.

Figure 15. Graphical analysis of a single particle is shown in Figure 16. This particle's diameter (full width at half maximum) is ~0.75 nm. The natures of these features need to be investigated; however, it is assumed that these features might have arisen from some sort of inclusions in the grown SiGe layer.

An example of nanometer size features that are seen in the SiGe

layer is shown in Figure 15 by taking a single layer from Figure 13. Figure 16 shows a graphical analysis of a single particle of size ~0.75 nm. The origin of these nanoparticles is not clear yet; however, it is presumed that the small features seen in the image may be some sort of particulate inclusions. More investigation is necessary to establish the nature of different kinds of defects. Another example of a multilayer image is shown in Figure 17. The images by the current technology may be divided in to any number of slices per sample requirements; each slice may be inspected individually. This layer by layer capability is important for detection and identification of defects in a non-destructive fashion.

In summary, nanometer and sub-nanometer scale resolution are necessary to analyze current and future integrated circuits due to their ever increasing functional density. Terahertz reconstructive imaging with nanometer resolution in conjunction with terahertz time-domain spectrometry is indispensable tools for viewing and characterizing nanoscale features and defects, as demonstrated in the present paper. This technology is non-contact, non-destructive and requires virtually no sample preparation. In contrast, TEM, for instance, requires a lengthy and laborious sample preparation and yet not able to obtain layer by layer information (Figures 9-17).

Conclusions

In this paper, 3D, sub-surface imaging is accomplished by means of terahertz nanosensing via a reconstructive route in a non-destructive and non-contact fashion with virtually no sample preparation requirements. A dendrimer dipole excitation based CW terahertz source was used. Terahertz time-domain spectroscopy was used for interrogating materials both on the surface and in the sub-surface region. Fourier transform of the time-domain signal was used to compute the absorbance spectra of the samples under investigation. It was found that the absorbance spectra, up to 10 THz, yields unique signature for Ge and SiGe epitaxial layers, as well as for Si <001> substrate. For reconstructive imaging, gridding with inverse distance

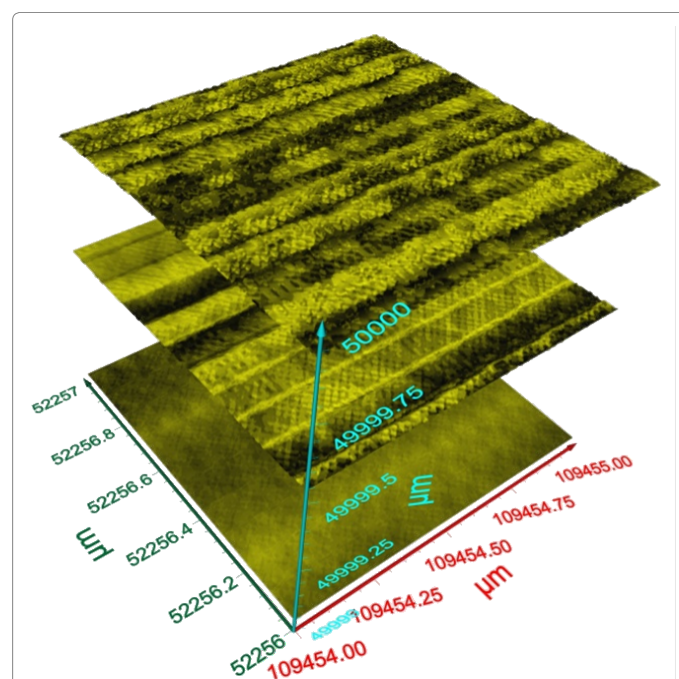


Figure 17: An example of layer by layer imaging. Here three different layers of sample D10 are extracted from Figure 12.

to power equations was utilized. Both 2D and 3D images were analyzed for unique identification of features and/or defects. It was found that the techniques deployed herein are effective for visualizing the lattice structure from a 3D image of the samples. Details of stacking fault have been observed with quantitative lattice dimensions. Layer thicknesses of epitaxially grown SiGe layer on Ge buffer and also of Ge buffer on Si wafer match with the TEM results for the same samples. Sub-nanometer particulates were observed in the SiGe layer; the origin of which require further investigations. The non-contact measurement system outlined herein may be implemented for quality inspection at the production line as well as for various process development purposes. It is notable that this is the first time a 3D lattice image has been produced for these samples. Unlike, a TEM image which shows only a single surface; the images of present paper are true 3D and allows inspection of the specimen on a layer by layer basis.

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