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# Growth study of GeTe phase change material using pulsed electron-beam deposition

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## ABSTRACT

Thin film growth of chalcogenides composition is an exciting field, owing to the interesting optical and electrical properties of them. In this work, a novel, physical vapor deposition (PVD) method called pulse electron-beam deposition (PED) experimentally studied to investigate the requirements for ablation of phase change materials (PCM). To evaluate the qualifications of the PED method, thin films of Germanium Telluride (GeTe) are grown and investigated by contemplating the surface morphology and the material composition. High-quality GeTe thin films with thicknesses from 30 to 200 nm, were successfully grown on top of silicon wafers at room temperature to demonstrate the ability of PED. To optimize the growth procedure, several growth parameters were thoroughly investigated, including background pressure, pulse energy, and growth temperature. A series of material characterization methods were adopted to study the GeTe material quality after the growth. These methods include field emission scanning electron microscope (FESEM), energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), and thickness profilometer. It was found that higher material growth rate can be obtained in lower background pressure (~ 2.6 mTorr), lower temperature (room temperature) but higher pulse energy (e.g., 15 kV). Besides, by increasing the target-to-substrate distance, the surface quality (e.g., smoothness) was improved substantially, but the growth rate decreased linearly. Finally, after the growth optimization, FESEM images revealed that the as-grown GeTe films were of high smoothness and uniformity. EDX analysis indicated that the compositions of the GeTe films were pressure dependent. Through the XRD spectrum, it is found that the as-grown GeTe films were amorphous. In order to convert them into crystalline formation, further post-treatment approaches (e.g., annealing) will be required.

## 1. Introduction

Phase change materials (PCM) are a class of materials in which their resistivity has a significant change due to transitioning back and forth between crystalline and amorphous states. In 1968, the first report on phase change material claimed that a special category of materials could change resistance drastically by applying electric field [1]. Phase change material was first used commercially in the 1990s for digital information storage, exploiting the material's optical reflectivity difference between the amorphous and crystalline phase [2,3]. Ever since the first article on PCM-based memory was published, researchers in this field have actively been working to discover new series of PCM materials and new ways to use their bi-stable characteristics for memory applications. Another noteworthy difference between the two states of PCM is the considerable change in electrical resistivity which can be several orders of magnitude. In the last couple of years, a new exciting application of PCM, low-loss and low-power switching, has been investigated [4,5]. The future of wireless communication will rely on the ability of radio frequency (RF) modules to accommodate multiple frequencies and functionalities. One approach to achieve reconfigurability is using RF switches to select between specified circuit blocks. Therefore, low-loss switches are crucial for high-performance operation. Among all Ge-Sb-Te (GST) material compositions, thin-film GeTe offers the highest contrast between its two stable states regarding electrical resistivity and very fast crystallization speed [6–8]. Although scandium-doped antimony telluride PCM, which has been recently reported for memory applications, has the lowest crystallization time [9], it has higher electrical resistivity at crystalline state than GeTe. Therefore, it was not considered for this work. More importantly, GeTe has the lowest resistance in the crystalline state, as well as the lowest crystallization temperature [8]. This means that a low loss RF switch with minor actuation power can be realized using GeTe material. State-

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of-the-art switches in current RF systems are almost exclusively silicon-based [10,11]; nonetheless, they have limitations. These switches need a constant supply of power to maintain a state. The advantage of the PCM-based switch over its silicon counterparts is that it does not require external power to maintain any of its states. Power is only required during the transition from one phase to another. In addition, PCM switches offer lower loss, which is important for the realization of the next generation of RF and wireless systems.

Currently, sputtering is the most popular deposition technique for growing GST and GeTe thin films [4,12,13]. Molecular beam epitaxy (MBE) [14] and pulsed laser deposition (PLD) [15–17] are two other commonly used growth methods. Nevertheless, each has limitations such as low growth rate, complexity, and ability to ablate a limited selection of materials. Pulsed physical vapor deposition (PVD) methods can precisely control the growth rate (sub-monolayer per pulse). Compared to sputtering, there is no need for considering the electrical properties of target materials, and smaller targets of expensive and highly pure material are required [15]. MBE uses distinct elemental targets to grow compound materials, and an ultra-high vacuum (UHV) chamber ($<10^{-9}$ mTorr), both increase the complexity and cost, but PVD methods utilize one compound target and do not need UHV for operation [14]. Moreover, both sputtering and MBE suffer from low deposition rates. PLD is the most studied and frequently used pulsed PVD methods that has been used to deposit GeTe thin-film layers. However, it has limitations regarding the laser beam reflection due to the plasma optically shielding the target. The shielding results in lower growth rate, which becomes even worse by the expansion of the plasma over time.

In this paper, a first-time demonstration of using a novel method for growing GeTe thin films is reported. This growth method is called pulsed electron-beam deposition (PED). The ablation mechanism in PED is similar to PLD, where the laser beam is replaced by concentrated electron beam. While PLD suffers from optical shielding of the target surface, the electron beam in PED is not reflected by the plasma, which results in improved efficiency. Also, there is no need for large, expensive excimer lasers and optical setups that are necessary for PLD [18]. PED was successfully established to grow materials like GaN [19], $CeO_2$ [20], $SnO_2$ [21], ZnO [22], and complex oxides [18]. This paper presents the first experimental study of using PED to grow chalcogenide materials. To demonstrate the PED capabilities, GeTe thin film, the most motivating PCM in the high-frequency applications is targeted. The primary motivation of this study is to demonstrate the advantages of utilizing PED method for growing complex composite thin-film materials.

## 2. Experiment

GeTe thin films were deposited on (100) silicon substrates at room temperature, by means of pulsed electron-beam deposition using a single 1 in., 99.99% GeTe target. The deposition process was carried out in a high vacuum system equipped with a channel-spark source (PEBS-20) from Neocera, Inc. Before starting the deposition process, the substrate was ultrasonically cleaned in acetone and isopropyl alcohol. After that, the wafer was placed underneath the GeTe target at a target-to-substrate distance varying from 3 to 8 cm. In the growth process, the deposition chamber was vacuumed down to a base pressure of $\sim 1 \times 10^{-6}$ Torr. The growth was conducted in an Ar environment with the background pressure varying from 2.6 to 5.5 mTorr. Since Ar is an inert gas and well suited for background deposition purposes [23], it was used as the background gas during deposition. The pulsed electron-beam source (PEBS) was operated from 11 to 17 kV at 5 Hz frequency with a total number of ~ 20,000 pulses. During the growth, both the target and the substrate were rotated to avoid damage to the target, and to improve the thickness uniformity of the films, respectively. The growth factors including E-beam generating voltage, target-to-substrate distance, background gas pressure, and the growth temperature were investigated, to find optimized growth conditions. After the growth, the thin films were systematically characterized using a stylus profilometer (Alpha-Step D500) and a high-resolution Zeiss Neon EsB FESEM for the surface morphology. Energy-dispersive X-ray spectroscopy (EDX) was employed to study the material composition of the GeTe films. Correspondingly, for the crystal structure analysis, a Rigaku powder X-ray diffraction (XRD) system was employed.

## 3. Results and discussion

During the PED growth process, both the E-beam parameters such as PED voltage and plume range, as well as the deposition parameters including background gas pressure and substrate temperature, control the quality of the deposited thin films. The impact of these factors on the GeTe thin film and growth rate was investigated and the results are presented in this section. Higher throughput and lower fabrication cost are results of a higher deposition rate. More importantly, a higher deposition rate results in a lower impurity concentration in the deposited material [24]. First, the energy required to ablate the GeTe target was optimized experimentally, so that the energy is higher than the minimum energy required to penetrate the target, and less than the energy that causes the plume range to be longer than the target-to-substrate distance. PED is a channel-spark discharge system, in which the applied voltage defines the electron beam energy emitted to ablate the target material [25]. Fig. 1 presents the growth rate as a function of PEBS voltage. Although higher beam energy results in a higher growth rate and enhanced throughput, the surface morphology is determined by the energy that the target material requires for evaporation. If the applied voltage is lower than the optimum, lead to low growth rate due to the lack of energy to ablate the material. This was found when 11 and 13 kV potentials were applied to the PED gun. To increase the growth speed, the voltage was increased to 15 kV, which resulted in a growth rate of 0.04 Å/pulse or 1.2 nm/min. This is a reasonable rate for GeTe deposition compared to PLD [14]. Raising the voltage to 17 kV resulted in a higher growth rate, but poor film quality, in terms of number and the size of particulates formed on the surface. These particulates are common in pulsed PVD methods [25–28]. The possible sources are emissions from the target and extraction after ablation in the gas phase [25]. Nevertheless, the size and number of these particulates drop when the substrate is placed at the end of the plume range. This is because the optimal target-to-substrate distance is adjacent to the plume range [29]. The plume range was measured by its effects on the deposited film in terms of surface roughness and growth rate. Overall, the optimized E-beam voltage was found to be 15 kV.

The plume range is an important E-beam parameter during the growth. The key features that define the plume range are the applied

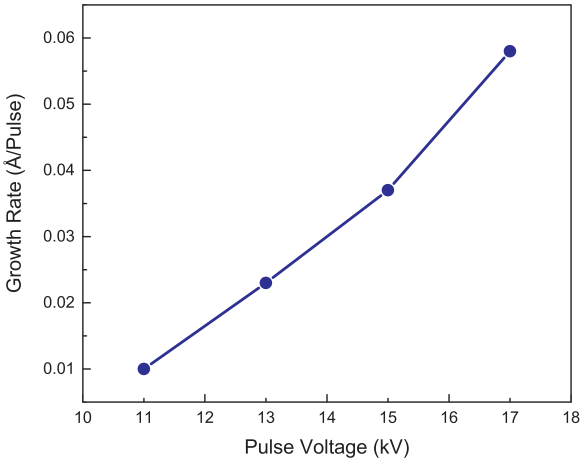

**Fig. 1.** Growth rate dependence on the applied PEBS at pressure of 5.5 mTorr and target-to-substrate distance of 8 cm.

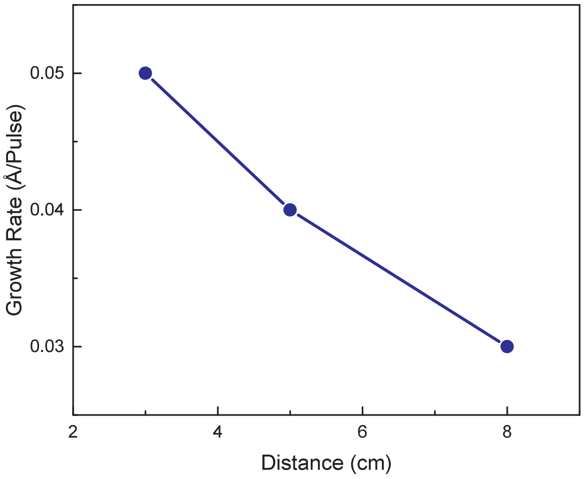

**Fig. 2.** Growth rate dependence on the target-to-substrate distance at pressure of 5.5 mTorr and PEBS of 15 kV.

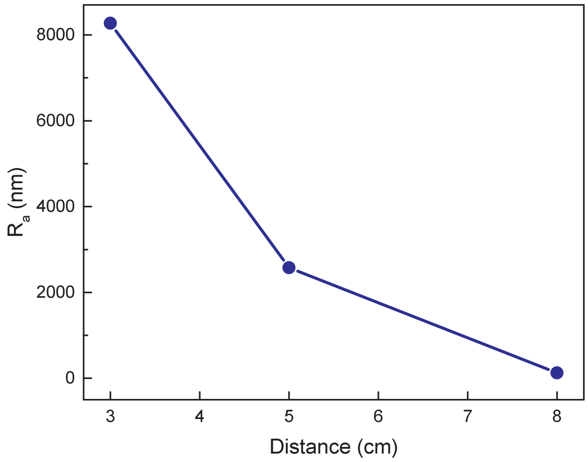

**Fig. 4.** Surface roughness as a function of target-to-substrate distance at pressure of 5.5 mTorr and PEBS of 15 kV.

PED voltage and the target-to-substrate distance. In our experiments, three different target-to-substrate distances were tested. The highest distance from the target that the chamber allows is 8 cm. Two extender substrate holders were used to grow thin films at 3 and 5 cm as well as 8 cm. Fig. 2 shows the average growth rate as a function of the target-to-substrate distance. The top view morphology results of 3, 5, and 8 cm are shown in Fig. 3 (a) to (c), respectively. In Fig. 2, the average growth rate has a linear and negative dependency on the target to substrate distance. Fig. 3 shows that, by decreasing the target substrate distance, some irregular particulates are formed on the deposited films. Although closer target resulted in a higher growth rate, the surface roughness was increased, and subsequently, the film quality reduced drastically. Lower the target-to-substrate distance resulted in larger particulates' sizes and densities. The arithmetical mean deviation surface roughness, $R_a$, was calculated from the measured height profile captured using a KLA stylus profilometer. The results shown in Fig. 4, confirm that the larger the distance, the smoother the surface. By increasing the target-to-substrate distance, a drastic reduction in the number and size of the particulates in the deposited film were observed, and the smoother surface was obtained.

Since the 8 cm distance resulted in the best surface morphology, and it is the largest distance that the chamber allows, the rest of the experiments were conducted at this distance. A higher PEBS potential (17 kV) was tested to confirm that 15 kV is the optimum PEBS voltage. Lower surface roughness results in higher performance of PCM in high-frequency applications. The purpose of this research is to achieve smooth film with no particulates larger than 200 nm, and with less than 1% of particulates covering the entire surface. The surface morphology of the as-deposited GeTe films, grown at 15 and 17 kV are shown in Fig. 5 (a) and (b), respectively. The results confirm that the 15 kV satisfies the desired requirements, and therefore it is the potential used for the rest of the experiments.

Ar background gas pressure between 2.6 and 5.5 mTorr was investigated for the GeTe thin-film quality. The PEBS requires at least 2.5 mTorr pressure for generating and sustaining the E-beam propagation. Pressures higher than 6 mTorr showed unbalanced materials compositions as well as low growth rate. Fig. 6 shows the average growth rate as a function of the background pressure. Decreasing pressure results in higher growth rates, which dramatically increases from 0.05 to 0.1 Å/pulse (1.5–3 nm/min) when the pressure changes from 3.5 to 2.6 mTorr. The cause is an increase in the mean free path at lower pressures. This higher growth rate results in improved purity in the grown material. Highly pure chalcogenide materials are required for less sheet resistance in the crystalline state, which is essential for high-frequency applications [13]. Oxidation of PCM during deposition is a common issue [13]. In absence of ultra-high vacuum (UHV), high growth rate reduces PCM oxidation. The oxygen content in the as-deposited GeTe films decreased from 3.5% to 0.6% by increasing the deposition rate from 0.05 to 0.1 Å/pulse.

Another challenge in the PCM thin film growth, which determines the sheet resistance, is reaching the proper ratio between material concentrations in the deposited thin film [30]. GeTe (50:50) has the lowest sheet resistance of the GST family of PCM. Previous research on GeTe materials showed that a slight change in the ratio of the elements does not have a considerable variation in the PCM properties [31]. Fig. 7 illustrates the impact of the background pressure on the ratio

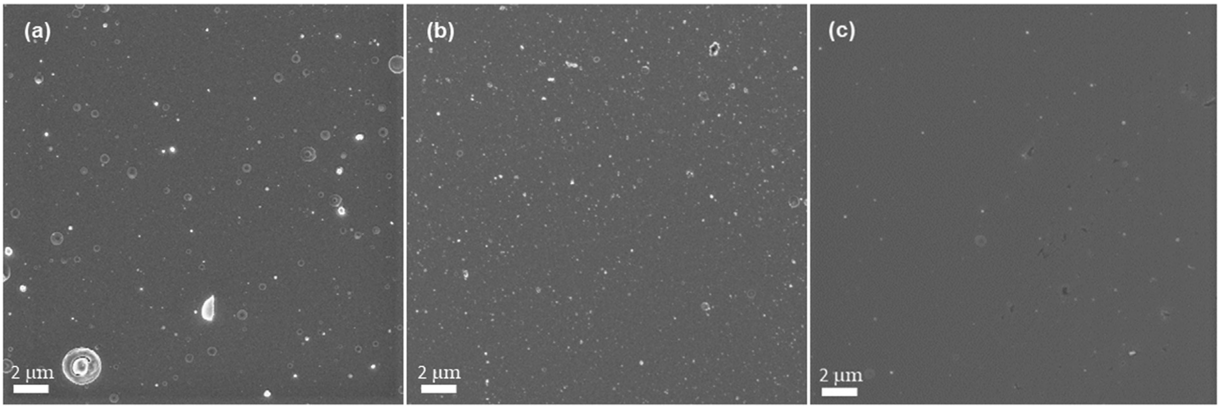

**Fig. 3.** Top SEM view of GeTe thin films deposited at (a) 3 cm (b) 5 cm and (c) 8 cm target-to-substrate distances at pressure of 5.5 mTorr and PEBS of 15 kV.

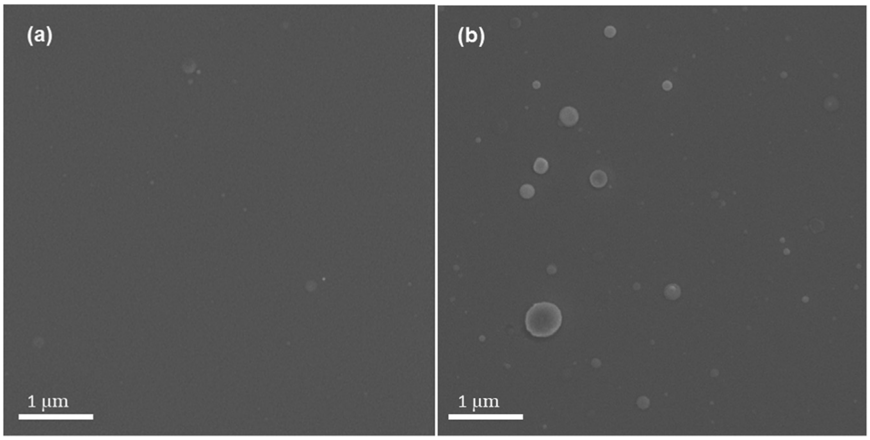

**Fig. 5.** Top SEM view of GeTe thin films deposited at (a) 15 kV and (b) 17 kV PEBS potentials at pressure of 5 mTorr and target-to-substrate distance of 8 cm.

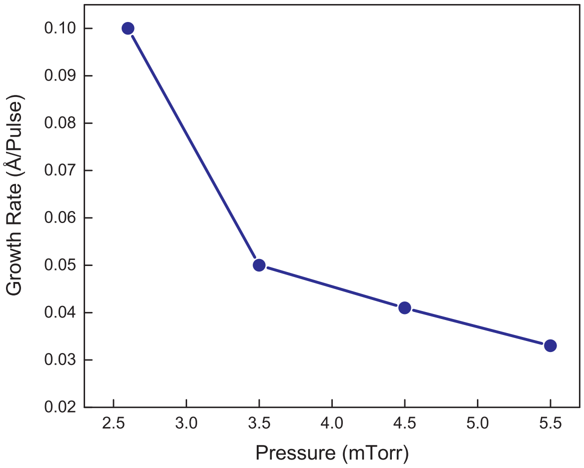

**Fig. 6.** Growth rate dependence on the background pressure at PEBS of 15 kV and target-to-substrate distance of 8 cm.

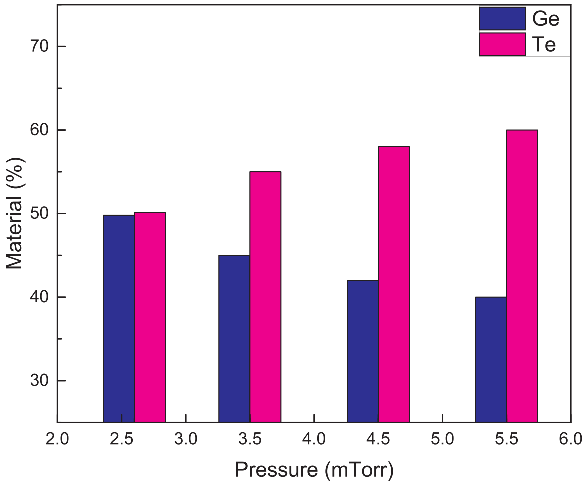

**Fig. 7.** Germanium and Telluride ratio dependence on the background pressure at PEBS of 15 kV and target-to-substrate distance of 8 cm.

between Ge and Te in the deposited material. Higher pressures result in telluride rich, thin films. This occurs because telluride rich clusters like $GeTe_2$ are formed and present in the plasma. These molecules can be deposited on the substrate at higher pressures [14]. The ratio between elements in the composition of Ge:Te can be controlled by reducing the pressure. Based on the trend of decreasing telluride and increasing germanium, the 2.6 mTorr provides $Ge_{50}Te_{50}$, which is the exact 50:50 stoichiometric composition desired. The error in EDX measurements was 2 Sigma, which means two standard deviations away from the mean in a normal distribution or 95.4% accuracy. To reduce the error, every sample was measured several times on different areas. The variation in the measured elemental ratios is around 0.5%, and the reported values in Fig. 7 represent their averages.

Amorphous and crystalline are two stable phases of PCM at room temperature. Epitaxial growth of materials can be performed at high temperatures to reach single crystalline thin-films material [32]. On the other hand, PVD methods are generally used to deposit PCM thin films at room-temperature [33,34]. In this study, the growth of the GeTe thin films, using the PED method, was performed at room temperature. Fig. 8 shows the XRD pattern for the GeTe thin film as deposited. The broad crest on the three-major crystalline GeTe materials' peaks ((003), (021) and (202)) verifies the amorphous structure of the as-deposited GeTe thin film. Post-annealing methods are commonly used to transform the amorphous as-deposited PCM material to a crystalline state [35,36].

Overall, the optimized growth parameters for depositing thin-film GeTe with PED system are summarized in Table 1. These conditions lead to smooth and uniform GeTe thin films deposited with the PED method.

## 4. Conclusion

In this study for the first time, pulsed electron-beam deposition has

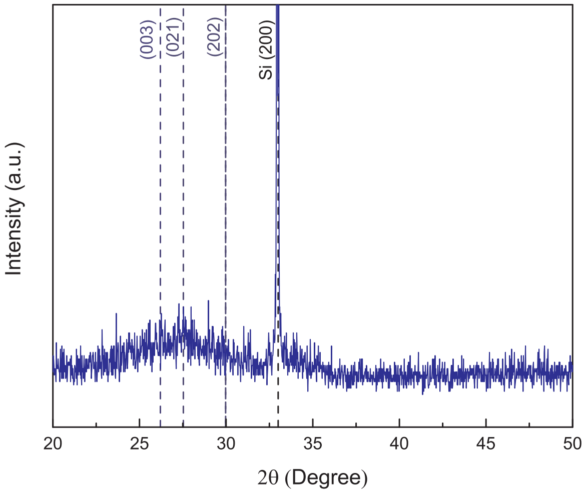

**Fig. 8.** XRD pattern of the as-deposited GeTe film.

**Table 1**
PED deposition parameters.

| Parameter | Value |
|---|---|
| Voltage | 15 kV |
| Target-to-substrate distance | 8 cm |
| PED frequency | 5 Hz |
| Chamber base pressure | 1 μ Torr |
| Deposition pressure | 2.6 mTorr |
| Deposition temperature | room temperature |

been employed to grow thin films of chalcogenides composition, and its capability to grow high-quality PCM thin films was investigated. GeTe thin films, the major PCM in the high-frequency utilization, have been successfully grown on silicon (100) substrates at room temperature. The quality of the deposited film was defined by the desired physical properties, such as smoothness and uniformity of the surface and the fundamental elements ratio of the material. The surface roughness of the films was defined by the plume range, which itself depends on the PEBS and target-to-stage distance. Background gas pressure determines the growth rate and the ratio between the two elements (Ge and Te). XRD profile was used to show that the as-grown GeTe thin film at room temperature was amorphous in configuration. Future work will include the crystallization of the amorphous GeTe thin films utilizing post thermal annealing and incorporation into high-frequency switches. The advantages of PED over other deposition methods, such as sputtering and PLD, make it a suitable alternative growth method for amorphous chalcogenides, as demonstrated by the growth of high-quality GeTe thin films. These materials offer exciting electrical properties for future RF applications such as agile communications and radar systems.

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