# Thin films of $Ge_2Sb_2Te_5$ prepared by pulsed laser deposition, properties and plume particles analysis

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## ABSTRACT

The pulsed laser deposition (PLD) was used for $Ge_2Sb_2Te_5$ (GST) thin films preparation. The process of ablation and clusters formation in PLD plume was studied. It was found that laser ablation of GST leads to the formation of many single charged particles (clusters) of Ge, $Ge_2$, Te, $Te_2$, $Te_3$, $TeGe_2$, $Te_2Ge$, $(GeTe)_2$, $GeTe_3$, $SbTe_2Ge$, and $Sb_3Te$ that were well identified. The formation of some higher mass clusters like $Ge_3Te_4$, $(GeTe)_5$, $Ge_2Te_8$, $Ge_9Te_4$ were also found. The composition of the clusters was proved via computer modeling and isotopic envelopes analysis. The thin films of GST prepared by PLD method and their basic physical properties were determined. They are similar to the properties of films prepared by other methods.

**Keywords**: laser desorption; TOF Mass Spectrometry; clusters, pulsed laser deposition, Ge-Sb-Te, thin films, properties.

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## 1. INTRODUCTION

Chalcogenide materials are widely applied as active layers of optical memory discs (DVD), as well as they are under intensive research targeted to development for alternative types of memories (Phase-Change RAM (PC-RAM) or Chalcogenide RAM (C-RAM)) to commercially successful semiconductor FLASH memories [1]. Both optical and electrical data recording is based on reversible phase transformation between amorphous and crystalline phases [2], caused by laser or electrical pulses application. Detection of the data is based on contrast in reflectivity/resistance between amorphous and crystalline state. The process is complex and many models have been suggested [3, 4, 15-21] to explain the changes of the structure during phase-change transition of GST films but mechanism of the process is still not fully understood.

Dominant compositions of active materials for their application in phase change memories include pure and doped Ge-Sb-Te, Sb-Te, or Ag-In-Sb-Te systems. The $Ge_2Sb_2Te_5$ (GST) thin films are finding application in electronics for data storage media including future non-volatile memories [3-7] and thermoelectric energy conversion [8]. GST can be synthesized as nanowires [9] and deposited as nano-films by various methods, by RF or DC magnetron sputtering [10], metal organic chemical vapor deposition [11], thermal evaporation [12], also by deposition from solutions [13], and by PLD of the GST bulk material [14].

However, the processes in plasma during laser ablation of GST are not well known or better to say it is not known at all.

The aim of this work was to better understand the processes during GST ablation and the processes in plasma plume. In order to resolve better the stoichiometry of the clusters formed from GST, laser ablation of the germanium, antimony, GeTe and $Sb_2Te_3$ was also studied. The PLD method was also used for thin films preparation and basic properties of the films were determined.

## 2. EXPERIMENTAL

Elemental Ge (LACHEMA Brno, 99.999%), Sb (Res. Inst. of Metals, Panenske Brezany, 99.9999%) and Te (KOCH-LIGHT Labs., 99.999%) were used for synthesis. Binary bulk GeTe and $Sb_2Te_3$ and ternary GST were prepared

from the constituent pure elements in sealed evacuated ampoules ($1x10^{-3}$ Pa, 30 min.). The synthesis was performed in a rocking furnace (800-950°C, 24 hrs).

The PLD method was used for GST thin films preparation. The PLD set-up consisted of a vacuum chamber ($p=1.4x10^{-4}$ Pa) and optical laser system. KrF excimer laser (Lambda Physik COMPex 102) was used to produce 248 nm light with constant output energy of 300 mJ/pulse, with pulse duration of 30 ns and a repetition rate of 20 Hz. Energy density of the laser beam on the target was varied from 0.5 to 1 $Jcm^{-2}$. The laser beam hit the bulk chalcogenide target at an angle of 45°. Target and substrate were rotated and their distance was 5 cm, substrate temperature ~ 40 °C. Thin PLD films were deposited on silica glass substrates from the pellets with composition of stoichiometric GST.

Bulk samples were used for Matrix Assisted Laser Desorption and Ionisation Time Of Flight Mass Spectroscopy (MALDI TOF MS). Prior to the mass spectrometric measurements, the small amount of either germanium, antimony, GeTe, $Sb_2Te_3$ and/or $Ge_2Sb_2Te_5$ was placed on a MALDI instrument sample target by an adhesive band.

The mass spectrometric experiments were performed using an AXIMA CFR (Kratos Analytical, Manchester, U. K.). The TOF instrument was equipped with a nitrogen laser (337 nm, repetition mode, frequency 10 Hz, pulse width 3 ns, Laser Science Inc., Franklin, MA, USA). The maximum laser pulse energy was 180 mJ.

Each mass spectrum was obtained by accumulation of at least 500 or up to 1000 laser shots. The measurements were always done in positive and negative either linear or reflectron modes. For external calibration of spectra measured in both positive and negative modes, carbon cluster ions formed from fullerenes and/or α-cyano-4-hydroxycinnamic acid ions were used.

Microanalysis of the bulk samples and films was carried out on an electron scanning microscope (JEOL JSM-5500LV) with energy dispersive x-ray (EDX) microanalyser IXRF Systems operated at 20 kV). Ellipsometric parameters psi, Ψ, and delta, Δ, (spectral ellipsometer, Variable Angle Spectroscopic Ellipsometry (VASE), J.A. Woollam Co., USA) were measured and optical parameters (index of refraction, n, extinction coefficient, k, thickness, d) calculated in 300-2300 nm spectral region. All ellipsometric, transmission, and depolarization data were analyzed simultaneously by VASE software.

Temperature dependence of sheet resistance was measured by Van der Pauw method [22] at average heating rate v = $2°C.min^{-1}$.

Raman spectra were obtained with Fourier Transformation (FT) micro-Raman spectrometer (Bruker) using λ = 785nm solid-state laser having an output power 20 - 36 mW. The resolution of the Raman spectrometer was 1 $cm^{-1}$.

## 3. RESULTS AND DISCUSSION

### 3.1 LD-TOF-MS (Laser Desorption-Time of Flight-Mass Spectroscopy) analysis of clusters of Ge, Te, Sb, Sb-Te and Ge-Te

Germanium [23], tellurium [24] and antimony [25] itself are forming many clusters during their evaporation. In plasma plume of Te, the positively and negatively charged clusters (the same as reported in [24]) were found, i.e. $Te_n$ (n =1-5).

Laser ablation and ionization of germanium (purity 99.9999 %) leads to the formation of large amounts of clusters, where small amounts of $Ge_m$ clusters are accompanied with a higher number of $Ge_mH_n$ clusters up to the m/z (m is for mass, z is for charge) closed to 1000 Da (Daltons). This is not surprising as metallic germanium is produced by reduction of $GeO_2$ by hydrogen and Ge-H is known to form solid films [26]. The highest mass of clusters were observed around 1190 Da, that corresponds to $Ge_{16}$ or to $(GeH_4)_m$ (m ~ 15) clusters. The similar behaviour was observed in the plume of pure antimony which is also known to form hydrides. Clusters Ge-H and Sb-H were not almost observed in the plume of GTS material.

Laser ablation and plume ionization of GeTe measured in positive linear or positive reflectron modes gave rich mass spectra with many peaks. An example of a part of the spectrum of GeTe is given in Fig. 1. The following clusters were identified: $Ge^+$, $Ge_2^+$, $GeTe^+$, $GeTe_2^+$, $GeTeH^+$, $Ge_2Te^+$, $Ge_3H_{18}^+$ (or $Ge_3H_{17}^+$), $(GeTe)_2^+$, and $GeTe_3^+$ species. Group of peaks around 740.8 Da was not identified up to now. Another group of the clusters around 1115.9, 1142.9, and 1168.7 Da were identified as $Ge_3Te_7$, an overlap of a mixture of $Ge_7Te_5^+$ with $Ge_{14}Te^+$ and an overlap of $Ge_9Te_4^+$ with $Ge_2Te_8^+$ cluster.

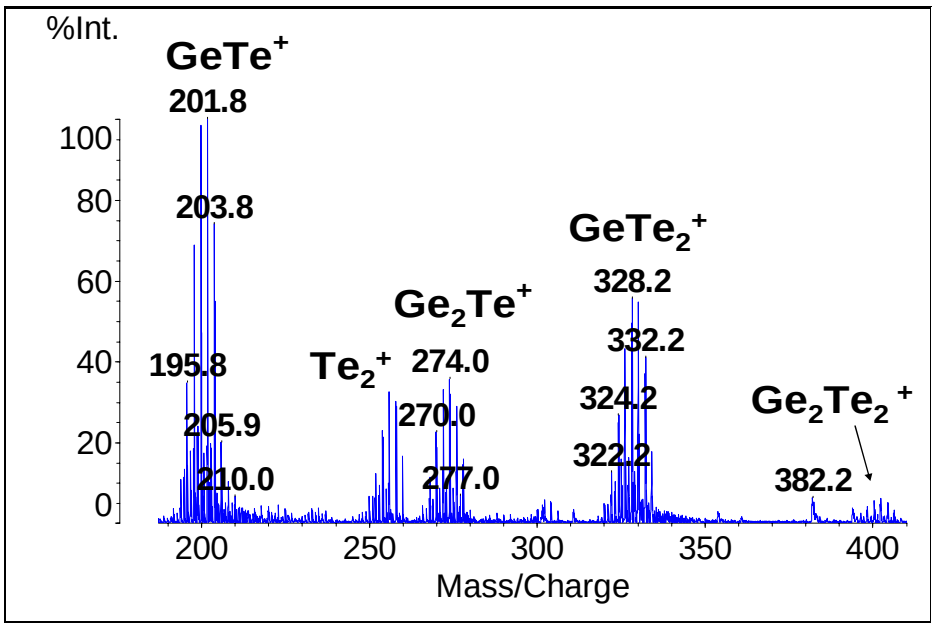


Fig. 1. Part of the mass spectrum of particles formed in the plume during the laser ablation of germanium telluride. Conditions: reflectron positive mode, laser power 180 mJ. The individual spikes correspond to combination of different isotopes of Ge and/or Te.

Laser ablation of antimony telluride ($Sb_2Te_3$) surprisingly lead to the formation of $Sb_mH_n$ clusters singly positively or negatively charged, like $Sb_2H_4$, $Sb_2H_6$, $Sb_2H_8$, and $Sb_4H_7$. Only some small intensity of signal corresponding to Te clusters was observed, and no binary Sb-Te clusters were found. The ionization of some particles during the ablation can be lowered (depressed) by the presence of the other components of the matrix (material). In such a case, no particles or their very low concentration, can be observed in the spectra. It probably pays for $Sb_n$ and Sb-Te clusters. The particles found are definitely present in the plume. The other particles, which were not identified, can be present as well, but if they are ionized with low probability, they are not seen.

### 3. 4 LD-TOF-MS analysis of thin films of $Ge_2Sb_2Te_5$

Thin films of GST were ablated using different levels of the laser energy. Singly charged clusters were observed in the negative and positive modes. Rather high energy of laser beam was needed to get higher clusters. The "best" spectra with higher resolution were obtained in reflectron positive mode and maximum laser energy. An example of the main part of the spectrum is given in Fig. 2.

The clusters identified were: $Ge^+$, $Ge_2^+$, $Te_2^+$, $Te_2Ge^+$, $GeTe_2Sb^+$ and $TeSb_3^+$. The signal of $Te_2^+$ cluster is not pure, it is superposed with some other cluster(s), which were not identified with certainty. It seems that there are some stiban clusters, most probably $Sb_2H_6$ cluster. The analysis of the overlapping peaks in the spectra concerning the overlay of $Te_2Sb^+$, $TeSb_2^+$ and $Te_3^+$ clusters in the range of m/z 350-380 Da are shown together with a computer model in Figures 3A and 3B. Low intensity peaks observed at around 579.2 and from 623.4 to 627.5 Da were not identified with certainty. The same clusters were observed in negative linear or negative reflectron modes and they were also singly charged. The intensities of their signals were different. However, no substantial difference was observed.

The experiments with matrices added to GST were yielding some clusters but mostly with small molecular weight. Mixed clusters which were obtained during laser desorption-ionization of GST were not observed here. Thus the experiments with the matrices were not done in extensive way.

Concluding this part, we can see that in plume created by laser ablation of GST, no clusters of $Ge_2Sb_2Te_5$ were observed, as well as no tellurium clusters higher than $Te_2^+$. There was just one ternary $GeTe_2Sb^+$ cluster observed. This is proving that the GST compound is mostly ablated to form just single elements and binary Te-Ge or Te-Sb clusters. Interesting is the observation of $TeSb_3^+$ cluster with high antimony content. This one is probably formed by some reactions

in plasma plume. Comparing the mass spectra measured in linear modes and in reflectron one, it can be also concluded that all the clusters are sufficiently stable and the prolongation of the flight time has no effect on the cluster type existence.

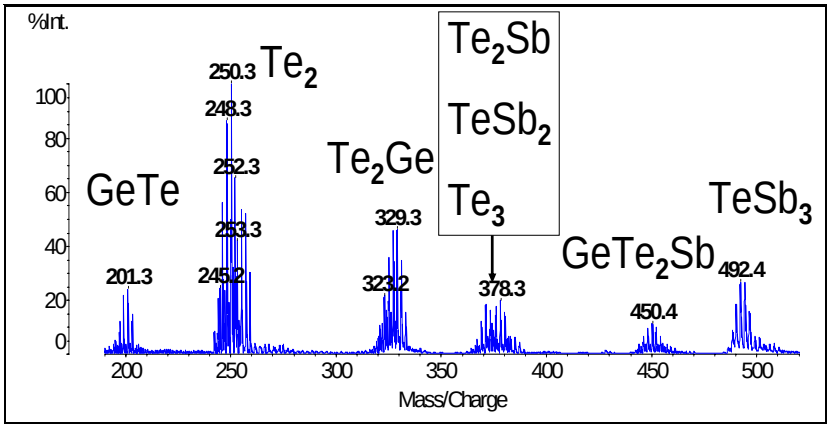


Fig. 2. Mass spectra of singly charged particles (clusters) formed in plume during laser ablation of $Ge_2Sb_2Te_5$ layer. Conditions: reflectron positive mode, laser power 180 mJ.

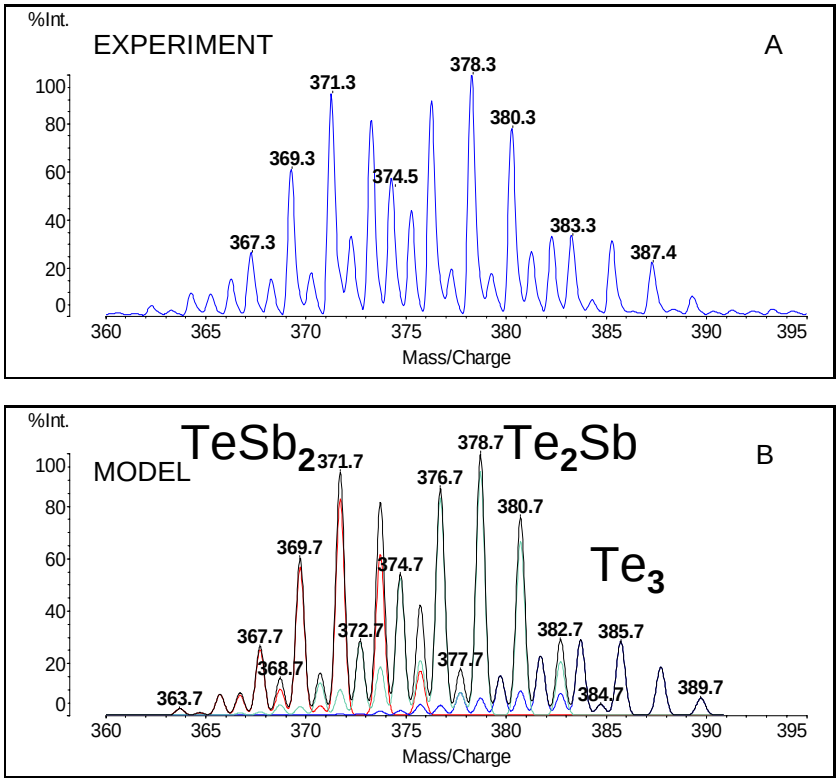

Fig. 3. The mass spectra of $Te_2Sb^+$, $TeSb_2^+$ and $Te_3^+$ clusters in the range of m/z 350-380 Da. (A) experimental spectrum; (B) computer model of the isotopic envelope concerning overlapping of $Te_2Sb^+$, $TeSb_2^+$ and $Te_3^+$ clusters. Conditions: reflectron positive mode, laser power 180mJ.

From the mass spectra of the plume formed by laser ablation of target it follows, that during the evaporation a strong dissociation of $Ge_2Sb_2Te_5$ is taking place with formation of Ge, $Ge_2$, $GeTe_2$, $GeSbTe_2$, SbTe, $SbTe_2$, $Sb_2Te$ and $Te_3$ charged or neutral particles. These particles can react together during the deposition process, forming highly disordered amorphous film. Only their heating above the crystallization temperature can lead to homogenization of the film with randomly occupied positions of "cations" (more positive atoms, Ge and Sb). It can be supposed that the full homogenization is possible only after re-melting of the layer or, probably after several cycles of phase changes crystalline-amorphous phase.

It can be supposed that similar dissociation and following homogenization takes place during preparation of thin films of GST by magnetron sputtering, and that similar sort of dissociation takes place during thermal or flash evaporation. The difference is probably only in the temperature of vapors, which is much higher for laser ablation [27].

### 3. 5 Properties of PLD films of $Ge_2Sb_2Te_5$

Thin films of GST were prepared by pulsed laser deposition technique. Three different deposition parameter sets were used (Table I.). Prepared films were amorphous as it is documented by XRD spectra presented in Fig. 4. The films had composition given in Table II.; their surface was decorated with small droplets (particulates) of the GST; their composition was slightly different from the composition of the starting target.

Tab. I. Parameters of pulsed laser deposition

| Sample no. | Target composition | Laser energy (mJ) | Pulse frequency (Hz) | Laser deposition time (min) | Film thickness (nm) |
|---|---|---|---|---|---|
| 1 | $Ge_2Sb_2Te_5$ | 300 | 20 | 10 | 190 |
| 2 | $Ge_2Sb_2Te_5$ | 200 | 20 | 9 | 140 |
| 3 | $Ge_2Sb_2Te_5$ | 150 | 20 | 10 | 125 |

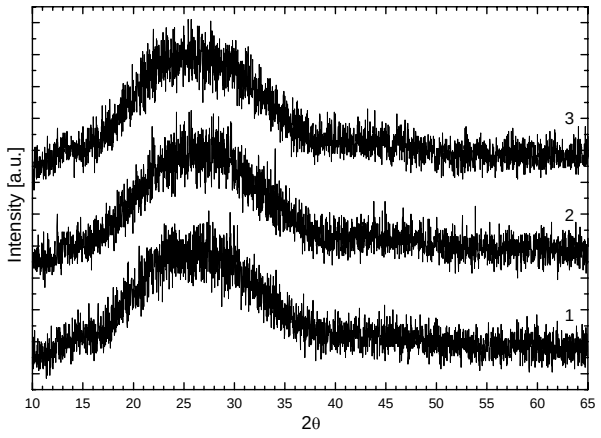


Figure 4

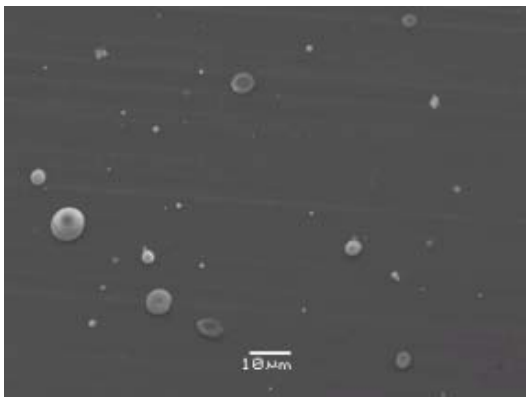


Figure 5

Fig. 4. X-ray diffraction patterns (XRD) of amorphous GST films prepared at condition listed in Table I. Curves numbers agree with sample nos. in Table I.

Fig. 5. Scanning electron microscope picture of the $Ge_2Sb_2Te_5$ film and its surface. Composition of the film and surface droplets is listed in Tab II.

Tab. II. Thin film prepared by PLD (sample no. 2, Tab I.) with laser pulse duration 30 ns.

| Composition | Ge [at. %] | Sb [at. %] | Te [at. %] |
|---|---|---|---|
| GST theoretical | 22.22 | 22.22 | 55.55 |
| GST thin film (Fig. 5) | 23.81 | 20.72 | 55.47 |
| GST droplets on surface of ablated film(Fig. 5) | 25.24 | 20.43 | 54.33 |

Thermal properties of studied films measured by DSC method (Fig. 6) revealed the glass transition temperature, Tg, in the range of 100-125°C; crystallization temperature, Tc, with onset around 175°C. Crystallization of the GST films prepared by PLD was studied also by resistivity of films measurement by four probes Van der Pauw Method (Fig.7), where crystallization is accompanied by sharp drop of resistivity near $T_c$. The change of film resistivity over several orders during heating and crystallization of the film was observed. It is in agreement with values obtained in other papers [10-13, 28].

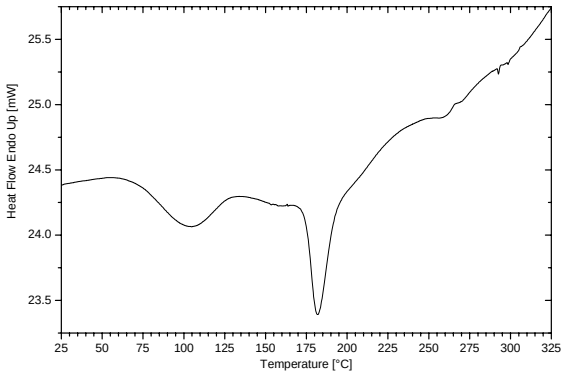


Figure 6

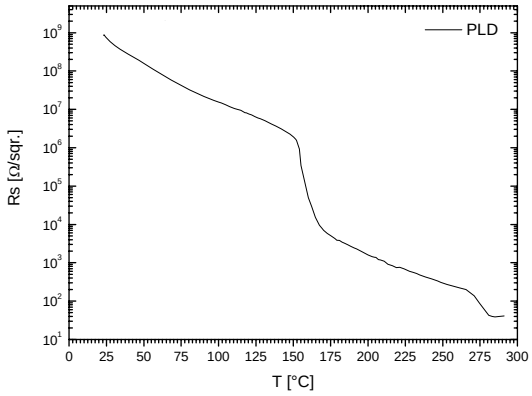


Figure 7

Figure 6 DSC curve obtained on PLD film (no. 2, Tab. I.) during heating scan at a rate 5K/min.

Figure 7 Van der Pauw sheet resistance measured on PLD GST film (no. 2, Tab. I.) during heating scan at a rate 2K/min.

Micro-Raman spectroscopy was applied to PLD films (Fig. 8) and in the spectra were identified vibration bands at 122, 139 $cm^{-1}$ and shoulder 161 $cm^{-1}$. Their assignment is not easy for GST. According to papers [29, 30], in which bands at 131, 147 and 166 $cm^{-1}$ were also found, they were assigned to vibration of particular structural units as follows: band at 131 $cm^{-1}$ ($GeTe_4$ unit), band at 147 $cm^{-1}$ (Te-Te unit) and band at 166 $cm^{-1}$ ($Sb_2Te_3$ unit). In a recent paper of Adrikopoulos [31] they found 3 bands in the Raman spectrum of GST crystal, one near 120$cm^{-1}$, second of lower intensity near 150$cm^{-1}$, and one, of very small intensity, near 276$cm^{-1}$. They attributed the first band (in analogy with GeTe vibrations) to the vibrations of corner-sharing tetrahedral units $GeTe_{4-n}Ge_n$ (n=0,1), the second band near 150$cm^{-1}$ to vibrations of $Sb_2Te_3$ structural units, the third band near 276$cm^{-1}$ to stretching Ge-Ge bonds vibrations. This band is of very low intensity and was not observed in our experiments. The detailed analysis of Raman spectra is under study.

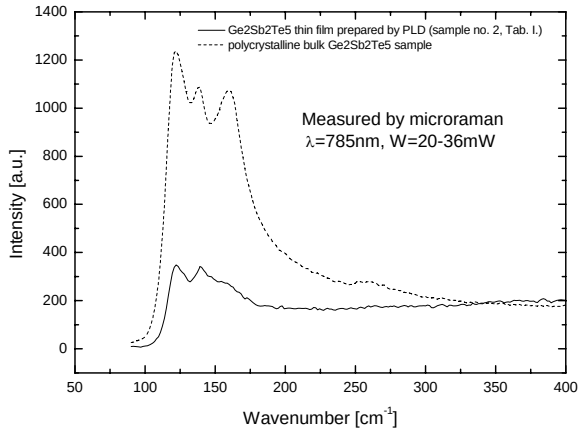


Figure 8 Micro-Raman spectra of the GST crystalline target and PLD films (no. 2, Tab I.)

It is well known that the composition of many telluride crystals and phase change memory materials can be changed in very broad ranges without any phase separation even in conditions close to thermodynamic equilibrium. Solid solutions and/or phases with a high density of different defects (substitutional [32] ones and vacancies [20]) can be formed even in equilibrium conditions [18 and Ref. herein]. To explain such a behavior, the formation of solid state solution with "antistructure" or "antisite" defects, like $Te_{Sb}$, $Sb_{Te}$, $Ge_{Te}$, $Sb_{Ge}$, $Ge_{Sb}$, where subscript describes the substituted atom, should be supposed. When the melt, or particles formed in plume during pulsed laser deposition, are cooled very quickly, the equilibrium is not reached and much more defects should be formed. Such a behavior is not common in classical semiconductors, but very common in so called "intermediate phases [18, 31], in semimetals or metallic alloys. In GST plays a role chemical similarity of atoms Sb, Te and Ge, their similar sizes, ($r_{covalent}$, nm: Sb= 0.141, Te= 0.137, Ge= 0.122); similar electronegativities: Ge =1.7, Sb =1.8, Te = 2.1) and similar bond energies (E, kJ/mol.: Sb-Te 195, Ge-Te 200, [18 and Ref. herein]). The high probability of formation of different defects enables the quick crystallization of many tellurides and phase change materials, including GST, because many atoms can occupy the positions of other atoms in crystal lattice. the presence of Ge-Ge vibrations bands found in the paper of Andrikopoulos supports the idea of antistructural defects formation [18].

## 4. CONCLUSION

In spite of the fact that layers of $Ge_2Sb_2Te_5$ are also formed by a laser ablation of bulk material, in plasma plume formed by laser ablation of GST using nitrogen 337 nm laser, clusters of $Ge_2Sb_2Te_5$ were not observed. Singly charged (both negative or positive) Ge, $Ge_2$, $TeGe_2$, Te, $Te_2$, $TeGe_2$, $Te_2Ge$, $(GeTe)_2$, $GeTe_3$, $SbTe_2Ge$, and $Sb_3Te$ clusters were well identified, as well as some higher mass clusters like $GeTe_4$, $(GeTe)_5$, $Ge_2Te_8$, $Ge_9Te_4$, which were difficult to distinguish. Thus, the $Ge_2Sb_2Te_5$ compound layer, produced by pulsed laser deposition, is most probably formed afterwards during cooling and crystallization process. They are not probably formed in the plasma plume. The formation of Ge-H and Sb-H clusters was also observed in MS spectra of GeTe and $Sb_2Te_3$ vapors during their deposition. Almost no Ge-H and Sb-H clusters were observed in spectra of laser desorbed GST material. The properties of GST films prepared by PLD are similar to those obtained by other evaporation method.

**Acknowledgements**
Authors thank to Ministry of Education, Youth and Sports of Czech Republic (Research Centre grant LC 523, grant MSM 0021627501 and MSM 00 216 224 11), to EC (FP6 project CAMELS IST-3-017406), to Czech Science Foundation (grant GA 203/06/1368) and the Academy of Sciences of the Czech Republic, project KAN101630651 for financial support.

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**Biography**

Professor Tomas Wagner has spent his professional carrier at University of Pardubice, Faculty of Chemical Technology, Department of General and Inorganic Chemistry and Research centre and became full professor in 2005. He was visiting scientist at several Universities e.g. University of Edinburgh, Scotland, University of Saskatchewan, Canada. The main topics of his research is solid state chemistry, mainly amorphous chalcogenides, their synthesis, characterization and study of their physico-chemical properties, structure and their potential applications. He was and is principal researcher or co-researcher of many projects at international and at Czech level. He organized and co-organized several international conferences and is co-editor and editor of special issues of international journals (J. Non-Cryst. Solids 2003, J. Phys. Chem. Solids 2007).