

Received 29 June 2018; revised 28 September 2018; accepted 8 October 2018. Date of publication 15 October 2018; date of current version 17 December 2018. The review of this paper was arranged by Editor K. Nojiri.

Digital Object Identifier 10.1109/JEDS.2018.2875927

Rigidity Enhancement of GeO₂ by Y-Doping for Reliable Ge Gate Stacks

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This work was supported by JSPS KAKENHI under Grant JP16H02331.

ABSTRACT The poor reliability of the GeO₂/Ge stack is improved by appropriate cation doping [e.g., Yttrium (Y)-doping] into amorphous GeO₂ as a result of the enhancement of the GeO₂ network structure stability. In this paper, we discuss the impact of Y-doping on structural modulation of GeO₂ on a Ge substrate in thermal treatment. By doping a small amount of Y into amorphous GeO₂, the crystallization of GeO₂ to α -quartz and α -cristobalite structures is efficiently suppressed without toughening the local Ge-O bond. This is direct evidence of rigidity enhancement of the GeO₂ tetrahedral network structure by cation doping.

INDEX TERMS Germanium, GeO₂, reliability.

I. INTRODUCTION

Germanium has been proposed as a candidate for high performance CMOS channel material due to its high electron and hole mobility values in bulk Ge. Recently, high performance Ge MOSFETs with a GeO₂/Ge gate stack formed by only oxidation have been demonstrated by many groups [1], [2]. These gate stacks have exhibited good initial performance even without forming gas anneal (FGA) but exhibited gate stacks with poor reliability (for example, trap generation by electric stress [3], [4]), which are very different from SiO₂/Si stacks. In SiO₂/Si stacks, it is well known that FGA is needed to achieve excellent initial properties, and the long-term reliability is excellent. On the other hand, we have demonstrated that the appropriate cation doping into GeO₂ substantially improves the long-term reliability in terms of the D_{it} generation at no cost to other initial performance degradations [5]. Here, the appropriate cation are, for example, Y, Sc or Al which do not form direct bond with substrate Ge. Furthermore, Y is one of the best dopants in terms of leakage current density at metal/doped-GeO₂/Ge stack [6].

We have proposed a rigidity model [7] to reasonably understand the difference between SiO₂ and GeO₂ and the impact of Y-doping on GeO₂ based on the stability of the amorphous network structure as described in the next section. However, such a doping effect on the structure of thin

amorphous GeO₂ film, for example, has not been investigated in detail from such a view point yet. In this work, we discuss the reasonability of the rigidity model through physical analyses of local and network structures of amorphous pure GeO₂, Y-doped GeO₂, and SiO₂ films in thermal annealing.

II. RIGIDITY MODEL FOR INITIAL PERFORMANCE AND LONG-TERM RELIABILITY OF GATE STACK

To describe the trade-off relationship between initial performance and long-term reliability based on both the single bond nature and network structure, we have proposed a rigidity model [7], which was inspired by the anticipation of insulator/Si interface initial defects based on the constraint theory of network structure [8]. We simply defined the rigidity as a product of the single bond strength γ and average coordination number N_{av} as the simplest model. As shown in Fig. 1, good initial performance and long-term reliability favor low and high γN_{av} , respectively.

In the model, the rigidity of SiO₂ is well-balanced and moderate considering the good initial performance and long-term reliability of the SiO₂/Si stack. Hence, the rigidity of GeO₂ is lower than that of SiO₂ because the Ge-O bond is weaker than the Si-O one [9], and rigidity enhancement is needed to improve the long-term reliability of the GeO₂/Ge stack. On the other hand, the doped-Y in the GeO₂ network

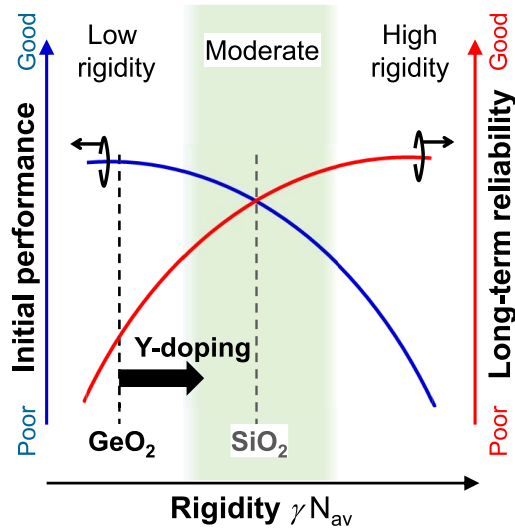


FIGURE 1. Initial performance and long-term reliability of MOS gate stack as function of rigidity γN_{av} , which is product of single bond strength γ and average coordination number N_{av} in rigidity model [6]. Initial performance and long-term reliability are in trade-off relationship. Comparing moderate (well-balanced) rigidity of SiO₂, rigidity of GeO₂ is relatively low due to weaker Ge-O bond. In this model, rigidity enhancement is required to improve long-term reliability of GeO₂ gate stack.

is expected to change the network structure with additional Y-O bonds due to a very high N_{av} of 5.6 in crystallized Y₂O₃. Namely, the improvement of long-term reliability in the Y-doped GeO₂/Ge stack might be mainly due to an increase of N_{av} . The detail of γN_{av} increase by doping and appropriate γN_{av} value for gate stack is semi-quantitatively discussed in ref. 7. From the next section, we experimentally analyze the actual Y-doping impact on local and network structures of GeO₂.

III. STABILITY OF AMORPHOUS GEO₂ STRUCTURE

Firstly, we investigated the structural stability of amorphous GeO₂ in the thermal annealing process. 270-nm-thick pure GeO₂ or Y-doped GeO₂ (YGO) films were stacked by RF-sputtering of GeO₂ or RF-co-sputtering of GeO₂ and Y₂O₃ on a chemically cleaned Ge(100) substrate, respectively. We also prepared a thermally grown SiO₂/Si stack as a reference. The pure GeO₂/ and YGO/Ge and SiO₂/Si stacks were thermally annealed in an ultra-high-vacuum chamber (base pressure: $\sim 10^{-8}$ Pa) at 660°C and 1200°C, respectively. The crystal structure was analyzed by XRD measurement. A Cu K α X-ray source was used for the XRD measurement.

Figure 2 shows the typical XRD profiles of thermally annealed pure GeO₂ and YGO stacks. The pure GeO₂ film clearly exhibited diffraction peaks assigned to the α -quartz and α -cristobalite structures in 5 min and longer annealing, which is in good agreement with previous work [10]. On the other hand, only the 1% (cation ratio) doped-YGO film does not exhibit any clear diffraction peaks caused by crystallization even in 15 min annealing. The small diffraction peaks appear again for the 24% YGO film, but this is due to another instability involved in phase separation,

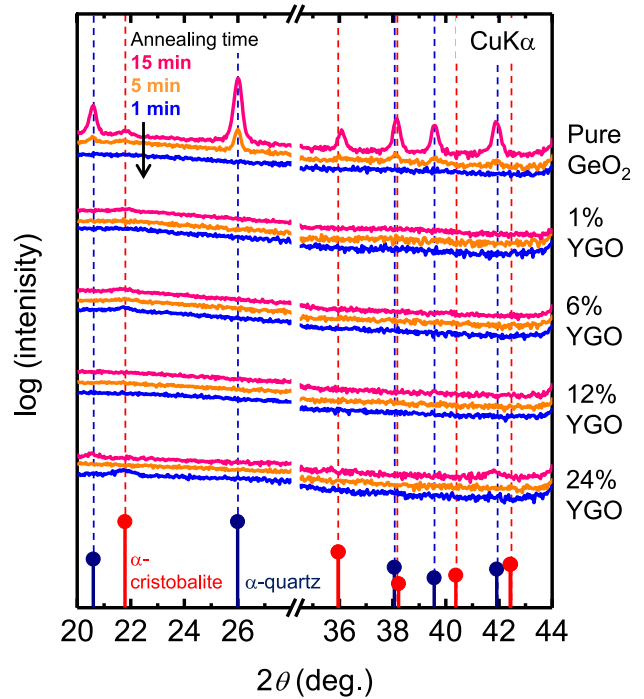


FIGURE 2. Typical out-of-plane XRD profiles of 270-nm-thick GeO₂/ and YGO/Ge stacks annealed at 660°C. Pure GeO₂ is crystallized into α -quartz and α -cristobalite structures in annealing, while 1, 6, and 12% cation ratio YGO do not exhibit clear diffraction peaks. Only 1% Y-doping is significantly effective to suppress crystallization of GeO₂ film. On other hand, 24% YGO film slightly crystallized into α -quartz structure with highly visible surface roughening. This might be due to another heavy doping instability resulting in spinodal decomposition.

resulting in decomposition with terrible surface roughening. In addition, the SiO₂ film does not exhibit any diffraction peaks caused by crystallization in the 1200°C annealing (not shown), though the annealing temperature of 1200°C (1473 K) for SiO₂ corresponds to that of 760°C (1033 K) for GeO₂ when the annealing temperature is normalized by the melting point of SiO₂ (1983 K) and GeO₂ (1388 K) in the absolute temperature scale. It is inferred that the amorphous network structure of GeO₂ has been improved a great deal and is like that of SiO₂ by the slight amount of Y-doping.

IV. LOCAL BOND STRUCTURE OF GEO₂

Secondly, we focused on the local GeO₂ structure. We analyzed the 270-nm-thick amorphous pure GeO₂ and YGO (1, 6, and 12%) films that had been annealed for 1 min at 660°C and the crystallized pure GeO₂ film that had been annealed for 15 min at 660°C by IR absorption and Raman spectroscopy measurements. The IR measurement was carried out in the vertical incident mode. The absorption peak around 870 cm⁻¹ observed in both the amorphous and crystallized GeO₂, as shown in Fig. 3(a), is assigned to the asymmetric stretching TO mode of Ge-O-Ge between neighboring GeO₄ tetrahedral units [11]. For Raman spectroscopy measurement, an Ar laser ($\lambda = 488$ nm) was used as an optical source. The Raman spectra were observed from 400 to

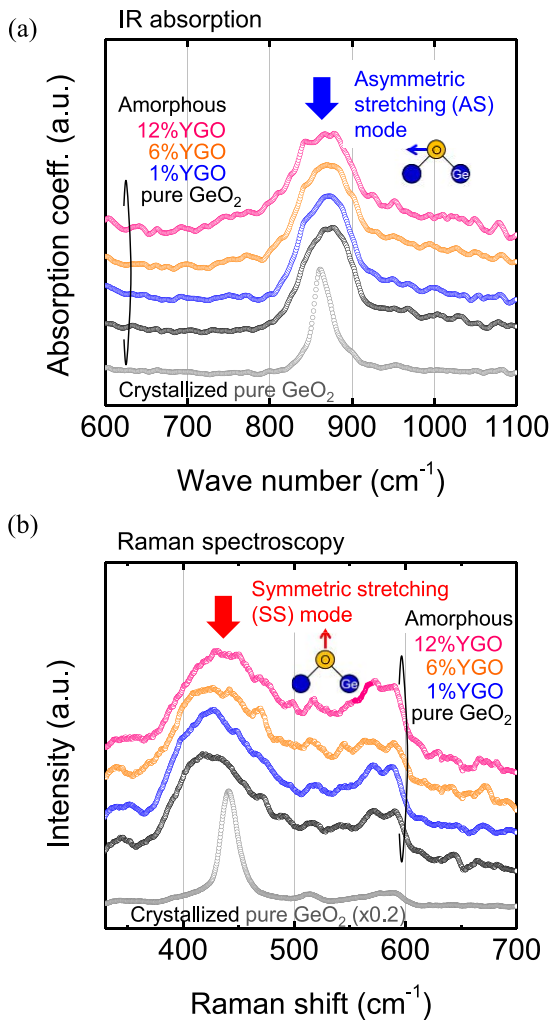


FIGURE 3. (a) IR absorption spectra and (b) Raman shift spectra of 270-nm-thick amorphous pure GeO₂ and YGO in annealing at 660°C for 1 min and those of crystallized GeO₂ in annealing at 600°C for 15 min. IR absorption peaks around 870 cm⁻¹ in Fig. 3(a) (blue arrow) and strongest Raman peaks around 430 cm⁻¹ in Fig. 3(b) (red arrow) are assigned to asymmetric and symmetric stretching mode of Ge-O-Ge bond between neighboring GeO₄ tetrahedral units, respectively. Obvious low energy shift of IR peak and high energy shift of Raman peak by crystallization of pure GeO₂ are reasonably understandable by narrowing of average Ge-O-Ge bond angle.

600 cm⁻¹ in both the amorphous and crystallized films, as shown in Fig. 3(b). The strongest Raman peak around 430 cm⁻¹ was assigned to the symmetric stretching mode of the 6-membered ring [12], [13] and low frequency edge of the band which originates in the breathing mode in the isolated molecule [14].

In pure GeO₂, peak energies and peak widths of the IR absorption and the Raman shift are characterized by the most likely Ge-O-Ge bond angle and its distribution. The peak energy of asymmetric stretching (AS) mode (IR absorption peak) and that of symmetric stretching (SS) mode (Raman peak) is clearly shifted down and up by crystallization, respectively. According to the well discussed phonon spectrum in AX₂ tetrahedral amorphous glass [14], [15],

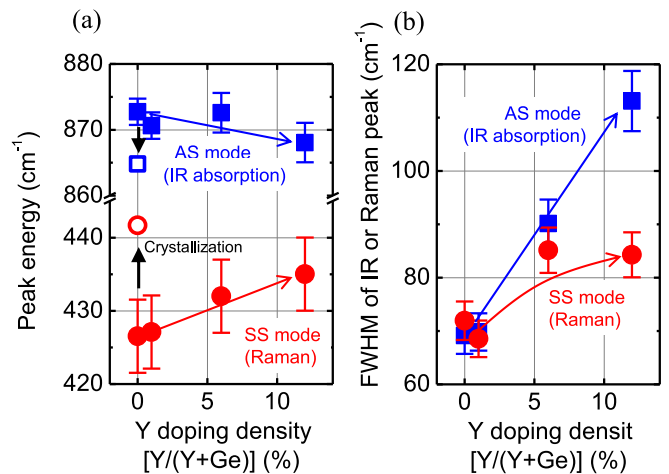


FIGURE 4. (a) Peak energy and (b) FWHM of IR absorption and Raman peaks as a function of doped Y ratio. The peak energy and FWHM were evaluated by fitting with a single peak. The AS and SS mode peaks do not exhibit obvious modulation by 1% doping. By increasing Y-doping up to 6% or higher, the peak energies are shifted in different direction and peak width is increased. The former effect is not explainable by Ge-O bond strength modulation but by slight narrowing of Ge-O-Ge bond angle. The later one is by increasing of bond angle distribution.

those peak shifts are reasonably understandable by the narrowing of the Ge-O-Ge bond angle in neighboring GeO₄ units [14]. The most likely Ge-O-Ge bond angle in crystallized GeO₂ is speculated to be lower than that in amorphous GeO₂ at 133° [16]. The narrowing of the peak width in IR absorption and Raman shift corresponds to the tightening of the Ge-O-Ge bond angle distribution, which is reasonably understandable based on the ordering of the local bond structure due to crystallization. From the above results, the crystallization of pure GeO₂ is described as the rearrangement and ordering of the local GeO₄ tetrahedral structure.

Next, we analyze the impact of Y-doping on local bond structure in amorphous GeO₂. The AS and SS mode peaks do not exhibit clear modulation with 1% Y-doping in terms of the peak energy and width, as shown in Fig. 4 (a) and (b), which suggests that the local structure of GeO₄ units is very little affected by 1% Y-doping. By increasing the Y-doping ratio to 6% or higher, the AS and SS mode peak energies are slightly shifted in the opposite direction, and the peak width is increased. According to the AX₂ tetrahedral amorphous glass model based on the central force between the A and X atoms [14], [15], the modulation of the A-X bond stretching constant induces the peak energy shifts of AS and SS mode in the same direction. Therefore, the slight peak shift is not mainly due to Ge-O bond strength modulation but to Ge-O-Ge bond angle narrowing, which is observed in pure GeO₂ crystallization. On the other hand, the broadening of the peak width corresponds to the increase of the Ge-O-Ge bond angle distribution. Thus, it is indicated that the significant stabilization of the amorphous GeO₂ structure by Y-doping shown in Fig. 1 is not mainly caused by toughening of the local Ge-O bonds.

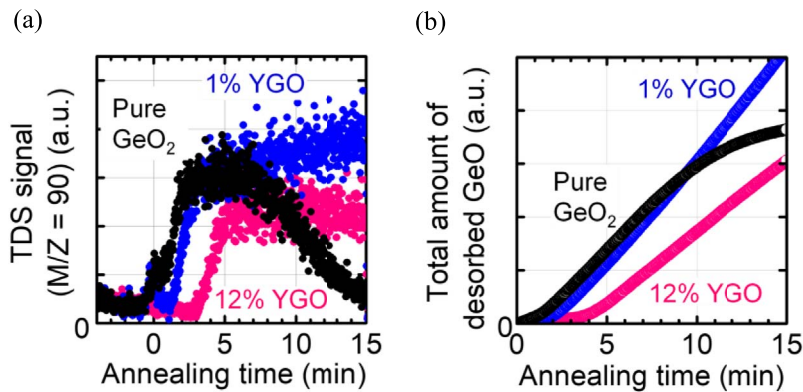


FIGURE 5. (a) TDS spectra of ⁷⁴Ge¹⁶O ($M/Z = 90$) desorption in 270-nm-thick pure GeO₂/ and YGO/Ge structure in annealing at 660°C which corresponds to GeO desorption rate. By increasing Y-doping density, initial GeO desorption is delayed. (b) Relationship between annealing time at 660°C and total amount of GeO desorption which is estimated from an integration of TDS signal shown in (a).

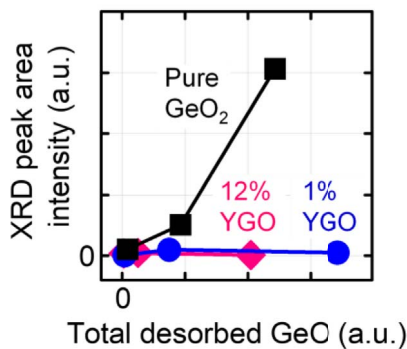


FIGURE 6. The XRD peak area intensity assigned to (101) α -quartz GeO₂ (observed around $2\theta \sim 26^\circ$) as a function of total amount of desorbed GeO shown in Fig. 4(b). It is obvious that YGO does not crystallize in highly GeO desorbed region, where pure GeO₂ is already crystallized.

V. CORRELATION BETWEEN AMORPHOUS STRUCTURAL STABILITY AND GEO DESORPTION

It is known that crystallization of GeO₂ is accompanied by GeO desorption [10], and Y-doping into GeO₂ effectively suppresses the GeO desorption [6]. Since GeO desorption from GeO₂/Ge stack is triggered by the O vacancy diffusion caused by GeO₂/Ge interface reaction [17], it is inferred that the destabilized GeO₄ tetrahedron network due to O vacancy diffusion might drive a further structural stabilization leading to the crystallized form and that the doped-Y in GeO₂ might stabilize the amorphous structure through perfect suppression of O vacancy diffusion in the thermal annealing. Actually, desorption of sub-oxide is not observed in SiO₂/Si stack in thermal annealing up to 1200°C at the lowest. Therefore, we also investigate the direct contribution of Y-doping to stabilization of an amorphous structure based on a correlation among Y-doping, O vacancies, and crystallization.

As an indicator of the O vacancy amount in GeO₂ and YGO films, we estimate the total amount of desorbed GeO in the annealing process. Fig. 5(a) shows the GeO desorption spectra measured by thermal desorption spectrometer (TDS). Thus, the total amount of desorbed GeO can be evaluated as

a function of annealing time by integrating the TDS spectrum. As shown in Fig. 5(b), Y-doping does not perfectly suppress the GeO desorption though the GeO desorption from the YGO/Ge stack starts with a time lag. Fig. 6 shows the XRD peak intensity assigned to the α -quartz structure of GeO₂ as a function of the total amount of desorbed GeO. It is clearly observed that YGO is hard to crystallize compared with pure GeO₂ even in the case that the total amount of desorbed GeO is the same. Thus, the result clearly indicates that the doped-Y stabilizes the amorphous structure in addition to delaying the GeO desorption.

On the other hand, the increasing of GeO desorption rate in initial stage in pure GeO₂/Ge is comparable to that in 1%-YGO/Ge, and then the GeO desorption rate from pure GeO₂/Ge obviously decreases with increasing annealing time as shown in Fig. 5(a). It suggests that the GeO₂/Ge interface reaction and/or O vacancy diffusion is drastically suppressed with progressing crystallization of GeO₂.

VI. DISCUSSION

These experimental results are reconsidered based on the rigidity model. As denoted in Section III, Y-doping is quite effective to suppress crystallization, which is described as a rearrangement of the GeO₄ tetrahedral network. This cannot be explained by perfect suppression of GeO desorption but by stabilization of the amorphous network structure, as shown in Section V. Therefore, it directly corresponds to the enhancement of the rigidity by Y-doping.

Furthermore, slight Y-doping into amorphous GeO₂ does not strengthen the local bond structure in GeO₄ tetrahedral units, as shown in Section IV. Thus, it is concluded that the rigidity enhancement of GeO₂ by Y-doping is mainly due to an increase of N_{av} by anchoring the amorphous GeO₄ tetrahedron network frame around the doped-Y as summarized in Fig. 7.

It is also reported that Y-doping in bulk germanate glass increases the glass transition temperature and bend strength [18], which is expected to be another aspect of the rigidity enhancement. Therefore, understandings of

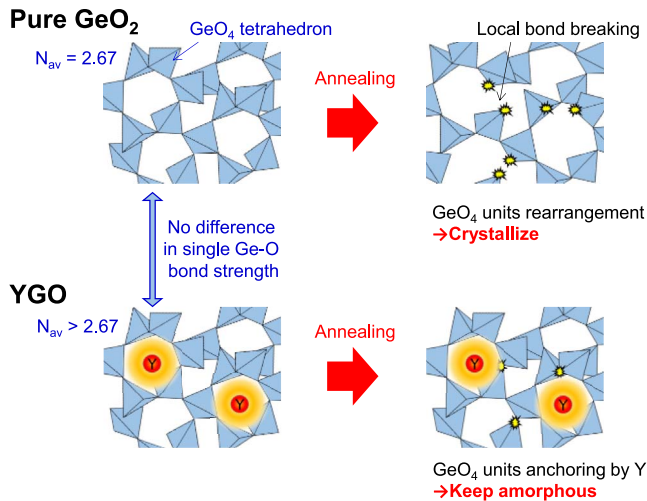


FIGURE 7. Schematic summary of impact of Y-doping on GeO₂ structure. Local bond strength of GeO₄ tetrahedral units in amorphous YGO is not different from that in amorphous pure GeO₂. However, amorphous network structure stability is different. It clearly appears when local bonds are partially broken by O vacancy diffusion as rearrangement and ordering of GeO₄ tetrahedral network. On other hand, Y-doping delays O vacancy diffusion but does not completely suppress it. Therefore, it is speculated that doped-Y may reinforce amorphous cation frame in GeO₂.

amorphous network structures control by doping in bulk glass seem to be effectively applicable to thin film in gate stacks, and it is concluded that the control of network rigidity of an insulator by appropriate doping is useful for designing gate stacks of new semiconductor materials as well as those of Ge.

VII. CONCLUSION

Crystallization of GeO₂ on Ge to α -quartz and α -cristobalite structures in thermal treatment is significantly suppressed by a slight amount of Y-doping, while the local bond structure is not significantly modulated by the doping. Therefore, it is understandable that toughening of the amorphous GeO₂ network structure occurs as a result of Y-doping. To realize a Ge gate stack with both good initial performance and long-term reliability, the design of a network structure based on the rigidity model will be a key to achieving high performance gate stacks.

ACKNOWLEDGMENT

The authors thank Dr. N. Hirashita, ESCO Ltd, for thermal desorption spectroscopy measurement of SiO₂/Si stack.

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