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Compression behavior of densified SiO_2 glass

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The compression behavior of fully densified SiO_2 glass has been measured up to 9 GPa at room temperature by using a diamond-anvil cell with a mixture of methanol-ethanol as a pressure medium. Optical-microscope observations clarify that there is remarkable agreement between the volume data on compression and decompression and therefore the glass behaves in an elastic manner. The compression curve can be expressed accurately enough by a Birch-Murnaghan equation of state with the zero-pressure bulk modulus $K_0 = 60.2$ GPa and its pressure derivative $K_0' = 4$ (fixed). Extrapolating this equation of state to higher pressures, the density of the glass merges with that of ordinary glass (i.e., former ordinary glass compressed to high pressures) at about 13 GPa. X-ray diffraction and Raman scattering measurements show that the first sharp diffraction peak and the main Raman band of the glass also merge with those of ordinary glass at the similar pressure range. These results suggest that the compaction of interstitial voids dominates in compression mechanisms of densified SiO_2 glass, similar to the case for ordinary SiO_2 glass. Together with available information from the literature, it is presumed that the permanent densification of SiO_2 glass takes place between 9 and 13 GPa at room temperature. The behavior of SiO_2 glass in its intermediately densified states is also discussed.

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I. INTRODUCTION

High-pressure behavior of SiO_2 glass has been studied extensively because it has attracted considerable attention in various fields of physical sciences, such as high-pressure physics, noncrystalline physics, applied physics, geophysics, etc. In particular, numerous papers on permanent densification have been published since its discovery in the middle of the 20th century.[1] Various SiO_2 glasses having a density of up to about 20% larger than ordinary glass have so far been synthesized as a function of synthesis pressure and temperature and other parameters,[2–4] suggesting the occurrence of continuous intermediate states between ordinary and fully densified glasses. High-pressure Brillouin[5] and Raman[6,7] scattering measurements, as well as molecular-dynamics simulations,[8] have suggested that densified SiO_2 glass behaves in an elastic manner (i.e., as an amorphous polymorph).

In this study the compression curve of fully densified SiO_2 glass has been measured on both increasing and decreasing pressure to confirm that it behaves in an elastic manner. The measured compression curve is compared with available data in the literature, such as bulk moduli of fully densified SiO_2 glass obtained by elastic-wave-velocity measurements,[9–11] compression curves of other amorphous and crystalline polymorphs of SiO_2,[12–18] and compression curves of intermediately densified SiO_2 glasses.[10,19] X-ray diffraction and Raman scattering measurements have also been carried out to obtain structural information of fully densified SiO_2 glass under high pressure. Compression mechanisms, intermediate states, pressure conditions at which permanent densification takes place, and related topics are also discussed.

II. EXPERIMENT

Densified SiO_2 glass was synthesized by using a belt-type high-pressure apparatus[20] at National Institute for Materials Science (Tsukuba, Japan). Synthetic ordinary SiO_2 glass in the form of a disk having a diameter of 6.0 mm and a thickness of 3.0 mm (or two disks having a diameter of 6.0 mm and a thickness of 1.5 mm) was used as a starting material. It was densified by heating to 873 K for 10 minutes at 10 GPa. Synthesis experiments were carried out several times. In all the experiments the recovered sample was broken into two large pieces (disks) and many small fragments. The density of large pieces was measured by the Archimedes method with distilled water as an immersion fluid. Small and thin fragments were confirmed to be the same as large pieces by Raman scattering measurements and used as a sample in diamond-anvil experiments.

High-pressure *in-situ* experiments were carried out with a diamond-anvil cell.[21] Measurements up to 9 GPa were carried out by using anvils having a 600-μm flat culet with a tungsten-rhenium gasket and a methanol-ethanol pressure medium (a 4:1 mixture of methanol and ethanol). This pressure medium has been reported to remain hydrostatic within the pressure range of this study.[22,23] The use of a helium pressure medium, which remains in a fluid state to higher pressures, was avoided because helium atoms may penetrate into interstitial voids of the sample.[18,24] Pressure was determined by the ruby-fluorescence method.[25] The culet of the anvil and the surface of the sample were not able to be focused simultaneously by an optical microscope throughout the experiments. This ensured that the sample, with an initial thickness of 60 μm, did not bridge between the anvils and was not affected by deviatoric stresses.

The volume change (V/V_0) was determined by measuring the change in size of the sample in optical-microscope images.[12,18,24] X-ray diffraction measurements were carried out by using an angle-dispersive method with 25 keV monochromatic x rays and an imaging plate detector at BL-18C of Photon Factory (Tsukuba, Japan). The exposure time was 8 and 5 hours for the sample and background, respectively. X-ray diffraction measurements at ambient pressure were carried out for a different sample (i.e., a large piece described previously). Raman scattering measurements were carried out by using the 488-nm line of an argon ion laser for excitation in a 150° light-scattering geometry and a 10× micro-optical spectrometer system consisting of a longpass filter, an imaging spectrometer, and a Peltier-cooled charge-coupled device. Raman spectra were corrected by measuring background.

III. RESULTS AND DISCUSSION

A. Compression curve

The pressure dependence of the density of densified SiO_2 glass is shown in Fig. 1. The zero-pressure density of the glass was measured to be $\rho_0 = 2.67$ g/cm^3 by the Archimedes method. This value corresponds to a 21% increase in density (cf. 2.20 g/cm^3 for ordinary SiO_2 glass), suggesting that the sample is fully densified. Errors in our zero-pressure density measurements may be as much as ±0.05 g/cm^3 and may not affect the discussion in this paper seriously. There is remarkable agreement between the data on compression and decompression, indicating that the sample behaves in an elastic manner. The zero-pressure bulk modulus was determined to be $K_0 = 60.2$ GPa, with its pressure derivative $K_0' = 4$ (fixed), by fitting a Birch-Murnaghan equation of state[26] to the volume data. This value is in good agreement with $K_0 = 60$–70 GPa, obtained by elastic-wave-velocity measurements.[9–11] The small difference may indicate that K_0' is slightly smaller than 4, assuming that a difference between isothermal and adiabatic moduli can be ignored. It has been reported that the compression curve of ordinary SiO_2 glass cannot be expressed accurately even by the fourth-order Birch-Murnaghan equation of state due to elastic softening.[12] It is worth pointing out that the compression curve of fully densified SiO_2 glass can be expressed accurately enough by the second-order Birch-Murnaghan equation of state.

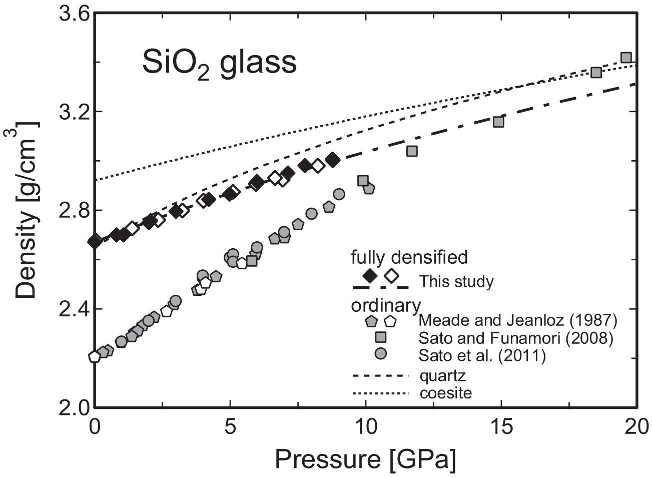

FIG. 1. Pressure dependence of the density of fully densified SiO_2 glass at room temperature. The pressure dependence of the densities of ordinary SiO_2 glass (Refs. 12 and 16–18) and crystalline phases of SiO_2, quartz and coesite (Refs. 13 and 15), is also shown for comparison. Black and gray symbols represent the data on compression, and white symbols represent the data on decompression. All the data for SiO_2 glass were measured with a mixture of methanol-ethanol as a pressure medium except for the data shown as squares (measured without a pressure medium). The dashed-dotted line represents the equation of state of fully densified SiO_2 glass ($K_0 = 60.2$ GPa and $K_0' = 4$).

The pressure dependence of the densities of ordinary SiO_2 glass[12,16–18] and crystalline phases of SiO_2, quartz and coesite,[13,15] is also shown in Fig. 1 for comparison. The data by Tsiok *et al.*[14] for ordinary glass measured with a toroid-type high-pressure apparatus, which are not plotted to simplify the figure, agree very well with those with diamond-anvil cells.[12,16–18] Although the compression curve of sixfold-coordinated SiO_2 glass is located outside of Fig. 1, its zero-pressure parameters have been reported to be $\rho_0 = 3.88$ g/cm^3, $K_0 = 190$ GPa, and $K_0' = 4.5$.[17] It seems reasonable that densified SiO_2 glass exhibits an intermediate compressibility between those of ordinary and sixfold-coordinated SiO_2 glasses. Densified SiO_2 glass also exhibits an intermediate compressibility between quartz and coesite. Because glasses are normally more compressible than their crystalline counterparts,[27] densified SiO_2 glass may be a coesite-like amorphous polymorph.

Compression curves have so far been reported for intermediately densified SiO_2 glasses.[10,19] The compression curves (and their extrapolation to higher pressures) of densified glasses with a different degree of densification in these reports appear to converge at around 8 GPa and 2.85 g/cm^3, while the compression curve measured in this study does not pass through this region. In both El'kin *et al.*[10] and Yokoyama *et al.*[19] measurements were carried out on decreasing pressure at a constant temperature (475–800 K) for the glasses densified by heating at high pressures. Strictly, they are therefore not compression curves but decompression curves. If materials behave in an elastic manner, their compression and decompression curves are the same. However, this is not the case. It is very likely that the structure (i.e., the degree of densification) of the glasses may have changed with pressure. In El'kin *et al.*[10] apparent compressibilities of the glasses are anomalously low at the highest-pressure range of their measurements, suggesting that structural transformations may have occurred at least in this pressure range. Moreover, in Yokoyama *et al.*,[19] since the compression curves were estimated based on elastic-wave-velocity measurements, errors must increase with increasing pressure in addition to the possibility of structural transformations. Nevertheless, the bulk modulus tends to increase with increasing the degree of densification, if the compression curves, including that of this study, are compared in a relatively low-pressure range. Therefore, intermediately densified SiO_2 glasses may also behave in an elastic manner, at least in a low-pressure range at room temperature.

The compression curve of fully densified SiO_2 glass gets close to the density of former ordinary SiO_2 glass compressed to high pressures[16,17] at about 13 GPa, as shown in Fig. 1. It is presumed that SiO_2 glass is fully densified by compression to this pressure (i.e., densification ends at this pressure). On the other hand, previous studies have clarified that densification begins at about 9 GPa.[5–7,10,12,14,28,29] Therefore, it is presumed that at room temperature under hydrostatic conditions the densification of SiO_2 glass takes place at a pressure interval between 9 and 13 GPa and then fully densified glass behaves as an amorphous polymorph up to the pressure where the structural transformation to sixfold-coordinated glass begins. The transformation to sixfold-coordinated glass has been reported to take place mainly at pressures between 20 and 35 GPa.[30] However, as shown in Fig. 1, the densities of SiO_2 glass measured by Sato and Funamori[16,17] are already larger than the extrapolated density of fully densified glass below 20 GPa. This may indicate that the transformation to sixfold-coordinated glass already begins in part at this pressure range, as pointed out by Brazhkin.[31] It is well known that nonhydrostatic stresses affect pressure conditions at which permanent densification takes place[2] (see the next section). Similarly, nonhydrostatic stresses may affect the transformation to sixfold-coordinated glass. Slight increases in coordination number can be seen below 20 GPa in Sato and Funamori[30] and Benmore *et al.*[32]

B. X-ray diffraction and Raman scattering

The x-ray diffraction patterns and the pressure dependence of the position of the first sharp diffraction peak (FSDP) of densified SiO_2 glass are shown in Fig. 2, and the Raman spectra and the pressure dependence of the position of the main Raman band are shown in Fig. 3. Raman spectra measured at ambient pressure before compression and after decompression did not show any significant changes, supporting the result of the previous section that fully densified SiO_2 glass behaves in an elastic manner. However, intermediate-range structure (i.e., network structure consisting of SiO_4 tetrahedra) changes with pressure. The position of the FSDP and that of the main Raman band shift to higher Q and higher frequencies with increasing pressure, respectively. These shifts indicate that the compaction of interstitial voids plays a main role in reducing the bulk-sample volume under high pressure.[18,24,33] Moreover, a so-called new peak[17,18,30–36] appears in the x-ray diffraction pattern at 9 GPa. This pattern shows good agreement with that measured at 10 GPa by Sato and Funamori[30] (see Sato *et al.*[37] to compare the two patterns in detail), suggesting that short-range structure (i.e., SiO_4 tetrahedron) is not altered significantly by applying pressure in this pressure range.

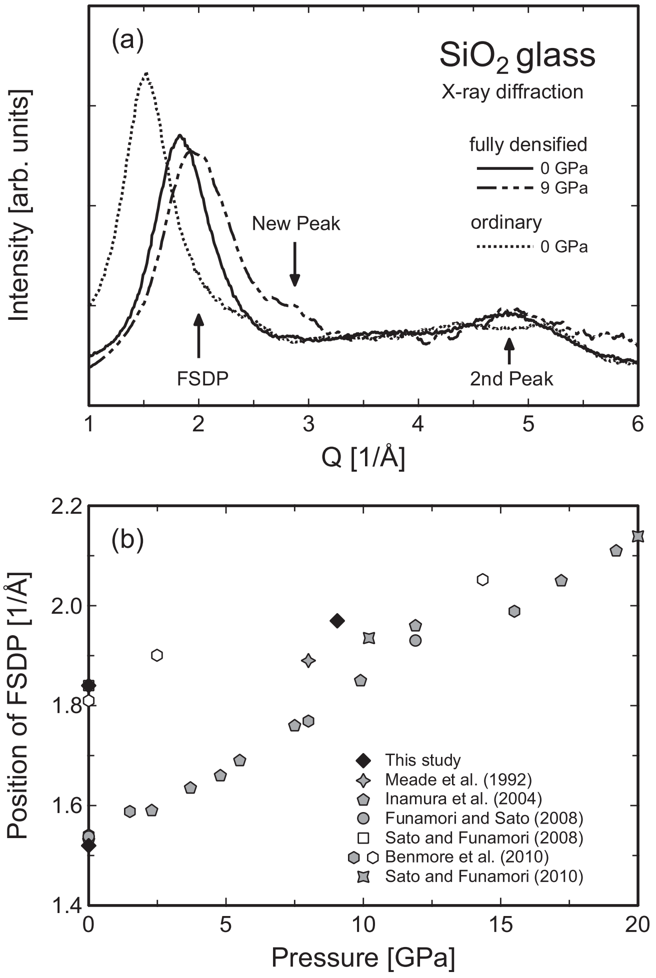

FIG. 2. X-ray diffraction patterns and the pressure dependence of the FSDP position of fully densified SiO_2 glass at room temperature. The pattern of ordinary SiO_2 glass at ambient pressure is shown for comparison. The FSDP positions read from the patterns in (a) are shown as black symbols in (b). Gray and white symbols in (b) represent the literature data on compression and decompression, respectively, which were measured in experiments on ordinary SiO_2 glass as a starting material (Refs. 16, 30, and 32–35).

The pressure dependence of the position of the FSDP [Fig. 2(b)] is quite similar to that of the density (Fig. 1). The data by Sato and Funamori[16] and Benmore *et al.*[32] measured on decreasing pressure from a pressure range of 30–40 GPa and those by Sato and Funamori[30] and Meade *et al.*[34] measured under conditions where the irradiation of intense white x rays has relaxed the structure may be assumed to be those for fully (or nearly fully) densified SiO_2 glass. This assumption is supported by the agreement between those studies and this study [Fig. 2(b)]. The position of the FSDP of fully densified SiO_2 glass is about 1.83 Å^{-1} at ambient pressure. The FSDP positions of former ordinary and fully densified SiO_2 glasses become indistinguishable at the pressure range where the densities of these two glasses merge, i.e., at about 13 GPa. This result is consistent with *in-situ* x-ray diffraction observations of the densification of SiO_2 glass by heating at high pressures by Inamura *et al.*[33] On the other hand, although the pressure dependence of the position of the main Raman band [Fig. 3(b)] is also similar qualitatively, the positions of the two glasses seem to become indistinguishable at about 7 GPa, in contrast to the case of the FSDP position and the density. This discrepancy, however, can be ascribed to the fact that the D_1 band affects the determination of the main Raman band position [see Fig. 3(a)]. Therefore, it is presumed that the main Raman band positions of the two glasses become indistinguishable at essentially the same pressure range as in the case of the FSDP position and the density. These similarities strongly suggest that the compaction of interstitial voids dominates in compression mechanisms of densified SiO_2 glass, similar to the case for ordinary SiO_2 glass.

The position of the main Raman band of densified SiO_2 glass is shown as a function of synthesis pressure in Fig. 4. These data were compiled by reading peak positions from spectra in the literature.[4–6,28,29,38–42] This figure shows that the peak position of fully densified SiO_2 glass is about 515 cm^{-1} and confirms that the sample measured in this study is fully densified. The black symbols represent the data

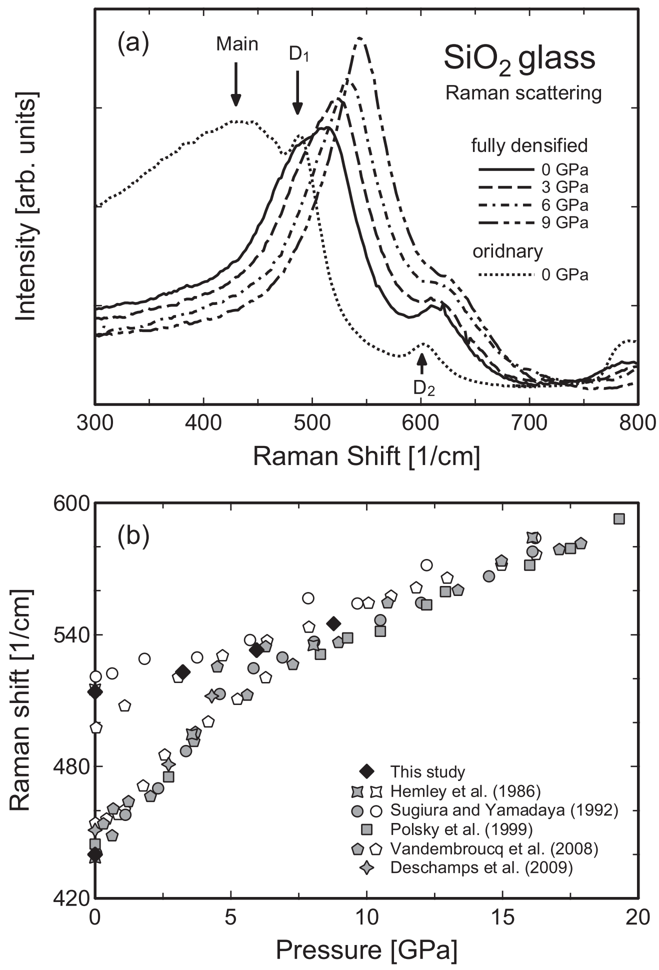

FIG. 3. Raman spectra and the pressure dependence of the main Raman band position of fully densified SiO_2 glass at room temperature. The spectrum of ordinary SiO_2 glass at ambient pressure is shown for comparison. High-pressure *in-situ* Raman scattering measurements were carried out on decreasing pressure from 9 GPa. The main Raman band positions read from the spectra in (a) are shown as black symbols in (b). Gray and white symbols in (b) represent the literature data on compression and decompression, respectively, which were measured in experiments on ordinary SiO_2 glass as a starting material (Refs. 7, 28, 29, 40, and 48).

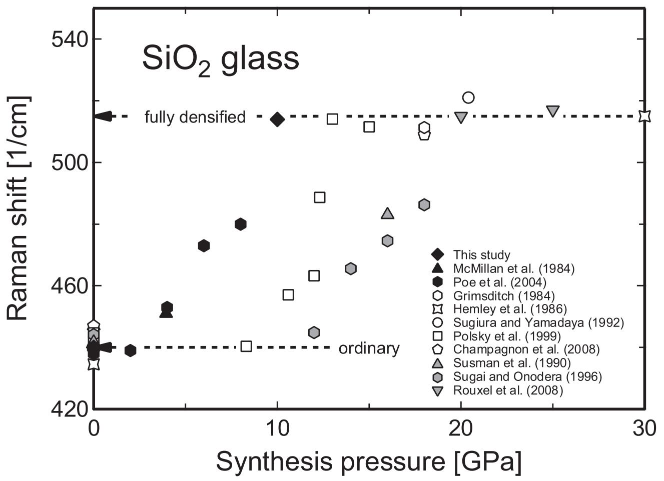

FIG. 4. Main Raman band position of densified SiO_2 glass at ambient conditions as a function of synthesis pressure. The position of ordinary SiO_2 glass is also shown for comparison. Black symbols represent the data of densified SiO_2 glass synthesized at high temperatures of 800–1000 K. Gray and white symbols represent the data of densified SiO_2 glass synthesized at room temperature under nonhydrostatic and hydrostatic conditions, respectively. It is well known that nonhydrostatic stresses not only hinder the densification at high pressures (as seen in this figure) but also promote it at low pressures, although Raman scattering measurements have not been reported. The data from the papers which show Raman spectra and details of synthetic conditions (Refs. 4–6, 28, 29, and 38–42) were complied by reading peak positions from the spectra.

of densified glass synthesized at high temperatures of 800–1000 K, including that of this study. These data suggest that the densification takes place between 2 and 10 GPa at 800–1000 K. Trachenko *et al.*[43] reported that the density increase by heating (former) ordinary SiO_2 glass to 700–730 K is 1.2–1.5% at 10 GPa, based on both experiments and molecular-dynamics simulations. This value is significantly smaller than 4–5%, which is the density difference between (former) ordinary and fully densified glasses at 10 GPa obtained directly from Fig. 1. Therefore, heating to 700–730 K is insufficient to synthesize fully densified SiO_2 glass at 10 GPa. The white symbols represent the data of densified glass synthesized at room temperature under (truly or nearly) hydrostatic conditions and, in this case, the densification takes place at a narrow pressure interval between 9 and 13 GPa. This is in remarkable agreement with the previous discussion based on the pressure dependence of the density (Fig. 1), the FSDP position [Fig. 2(b)], and the main Raman band position [Fig. 3(b)]. The gray symbols represent the data of densified glass synthesized at room temperature under nonhydrostatic conditions and suggest that nonhydrostatic stresses significantly affect pressure conditions at which permanent densification takes place. It is well known that nonhydrostatic stresses not only hinder the densification at high pressures (above 10 GPa) but also promote it at low pressures[2] (although Raman scattering measurements have not been reported and thus no data are plotted in Fig. 4).

No significant differences are found between the glasses densified at room temperature and at high temperatures. This is consistent with the conclusion of Inamura *et al.*[44] On the other hand, the glass may have a slightly different structure when decompressed from higher pressures, i.e., above 20–35 GPa where the transformation to sixfold-coordinated glass takes place. As for the D_2 band, when densified at higher pressures, the position shifts slightly to higher frequencies and the intensity increases slightly relative to the main Raman band [e.g., compare Fig. 3(a) in this paper and Fig. 2 in Hemley *et al.*[28]]. Moreover, the density and the FSDP position of the glass recovered from a pressure range of about 30 GPa have been reported to be 2.74 g/cm^3 and 1.84 $Å^{-1}$.[16]

It is likely that the reading of positions of asymmetric peaks, such as the FSDP and the main Raman band, could differ systematically among papers. In order to make a quantitative comparison, it is important to present patterns or spectra in a paper (or in its supplementary information). In this paper all the data in Figs. 3(b) and 4, except for those of Vandembroucq *et al.*,[7] were obtained by reading peak positions from spectra. Because the data of Vandembroucq *et al.*[7] at ambient pressure in Fig. 3(b) most likely come from the same spectra for the data of Champagnon *et al.*[6] in Fig. 4, the discrepancy is attributable to the difference in reading peak positions. Mukherjee *et al.*[45] reported the discovery of the first-order transition of SiO_2 glass with a density increase of about 20% at around 4 GPa and 950 K. It is worth pointing out that the x-ray diffraction pattern and the Raman spectrum of the high-density phase shown in their paper are completely different from those of densified SiO_2 glass shown in Figs. 2 and 3 in this paper.

C. Densification process

Vandembroucq *et al.*[7] considered densification as plastic deformation by pressure and, from this view point, explained the phenomenon at room temperature very well. In their model the plastic deformation takes place when compressed beyond the elastic limit and the network structure of SiO_2 glass changes with pressure by rebonding SiO_4 tetrahedra. Vandembroucq *et al.*[7] proposed that densified SiO_2 glass in a different state could be labeled with a maximum pressure applied to the glass as a fictive pressure. As discussed in the previous section, however, similar states can be achieved at a wide range of synthesis pressure due to the difference in temperature and/or nonhydrostaticity. Therefore, it is more appropriate to label simply with the zero-pressure density. According to this model, SiO_2 glass may behave as follows. In Fig. 1 the SiO_2 glass compressed to a pressure between 9 and 13 GPa is recovered from the pressure-density point to ambient pressure along its compression curve. The density of the glass recovered to ambient pressure is its zero-pressure density. When the glass is recompressed, it comes back to the original pressure-density point along the same compression curve. When the glass is compressed to a higher pressure between 9 and 13 GPa, further densification takes place and it behaves as a different glass having a new zero-pressure density. When the glass is compressed beyond 13 GPa, the densification is completed and it behaves as a fully densified glass with a zero-pressure density of about $2.67\ \mathrm{g/cm^3}$.

El'kin *et al.*[10] considered densification as phase transformation. In their model intermediately densified glasses can be synthesized because of the kinetic barrier (or hysteresis) of transformation. While plastic deformation is irreversible, densified SiO_2 glass can revert back to ordinary SiO_2 glass at high temperatures, as summarized by El'kin *et al.*[10] very well. At the same time, also in their model, the network structure of SiO_2 glass changes with pressure by rebonding SiO_4 tetrahedra, similar to the model by Vandembroucq *et al.*[7] Molecular-dynamics simulations[8,46] suggested that the rebonding causes modifications in the size distribution of the rings consisting of SiO_4 tetrahedra and therefore causes permanent densification. The transformation diagram proposed by El'kin *et al.*[10] is consistent with Fig. 1, which shows that the permanent densification of SiO_2 glass takes place between 9 and 13 GPa at room temperature. In the transformation region ordinary glass is densified by heating. In some papers[8,19] the volume decrease due to densification by heating was called as negative thermal expansion. This is misleading because the volume of densified glass does not increase with decreasing temperature.

It may be useful to discuss intermediate states in more detail by considering densification as phase transformation. In the case of crystals the occurrence of continuous intermediate states is impossible, although an intermediate phase, having a structure different from both low- and high-pressure structures, can be formed. The kinetics usually causes the coexistence two phases, i.e., low- and high-pressure phases, with various ratios. In the case of liquids intermediate states are often described as a mixture of low- and high-pressure components, with the ratio of the two components changing with pressure.[27,47] Moreover, intermediate states of liquids can be truly continuous. In both cases for liquids, however, an intermediate state does not behave in an elastic manner, because the kinetics usually does not matter and thus structure changes continuously with pressure. Therefore, the occurrence of continuous intermediate states and their elastic behavior may be characteristic of glass. The elastic behavior of the intermediate states has not yet been confirmed by experimental studies. Detailed high-pressure *in-situ* studies on the intermediate states will improve our understanding of the permanent densification.

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