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PLASTIC DEFORMATION OF AMORPHOUS SILICA : MODIFICATION OF THE RING STATISTIC

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Résumé - Nous montrons que la déformation plastique de la silice vitreuse en dessous de la température de transition vitreuse est caractérisée par une inhomogénéité du fluage. Celle-ci se manifeste par l'apparition de zones anisotropes qui sont examinées en microspectroscopie Raman. Une modification structurale est ainsi mise en évidence.

Abstract - We show that plastic deformation of amorphous silica below the glass transition temperature is characterized by a flow inhomogeneity. This appears as birefringent zones which are investigated by Raman microspectroscopy. A structural modification is thus put in evidence.

I - INTRODUCTION

Plastic deformation of amorphous materials below the glass transition is a widely studied topic. Most of the works concern amorphous metals and less covalent glasses. One of the main observations in metglass, for instance, is the inhomogeneity of the deformation : any deformation test (bending, traction, compression) induces slip lines /1/. This localized slip is often explained by assuming chemical or structural disordering /2/ without any experimental evidence of the modification due to plastic deformation. We report some experiments on silica glass that point out a drastic change of the sample after low temperature deformation and the connected structural modification. To put in evidence this alteration, we use the Raman microspectroscopy. With Raman spectra, we get some information on the modification of the medium range order : the two Raman active "defect" lines (D_1 : 490 cm^{-1} and D_2 : 605 cm^{-1} being linked to the concentration of fourfold and threefold rings of SiO_4 tetrahedra, according to Galeener's interpretation of the polarized Raman spectra in $v\text{-SiO}_2$ /3/.

II - EXPERIMENTAL RESULTS

Plastic flow

The flow apparatuses we use allow axial compression tests on small samples (about $(3\text{mm})^3$). We apply stresses up to 2 kbars with a sample temperature going from 1000°C to 1400°C . The flow rate $\dot{\epsilon}$ is obtained as a function of the temperature T and the compressive stress σ .

Above the glass transition temperature ($T_g = 1200^\circ\text{C}$), we find the well-known Newtonian behavior. Below T_g , the deformation law becomes non-linear /4/. Flow inhomogeneity appears at the same temperature ($T \sim 1000^\circ\text{C}$). Slip lines have never been observed but the samples exhibit birefringent zones. In order to see them, we cut a deformed sample in thin slices (thickness : $100\text{ }\mu\text{m}$) ; then, after polishing each face, the thin section is observed in a transmission microscope between crossed polarizers. When the sample has been deformed at a temperature above T_g , it remains isotropic but when the deformation takes place below T_g , numerous birefringent zones appear. The birefringence is roughly measured with a compensator : $\Delta n \sim 10^{-5}$. Photo n° 1, n° 2 and n° 3 show the same thin section but the direction of the deformation is each time different in every photo : birefringent zones remain obvious. It means that the optical axes are widely distributed, actually most of the zones have optical axes parallel to the stress direction or at 45° to this direction.

The directions of the polarizers are indicated by the arrow P, the stress direction by the arrow C.

Photo n° 1

The sample thickness is 100 μm . The spread zones and the crosses are obvious. Their quasi-regular disposal is more evident on the right side of the photo.

Photo n° 2

Sample thickness : 300 μm . We only see the spread birefringent zones.

Photo n° 3

Sample thickness : 100 μm . Some punctual birefringent zones appear. Spread zones are obvious especially alongside the sample.

Figure n° 1

Raman spectra of deformed and annealed silica glass.

- a) This sample has been deformed under 500 bars at $T = 1000^\circ\text{C}$. (deformation amount 10 %)

- b) This sample was in the same furnace as the previous one but it has been just annealed at $T = 1000^\circ\text{C}$ for the deformation time.

Two kinds of zones are obvious (photo n° 1) : spread zones and small birefringent crosses (rough size : 100 μm). Moreover these crosses seem to be regularly arranged. To our knowledge, such heterogeneity in the low temperature deformation of a covalent glass has never been observed. The sample remains amorphous : the birefringent zones are not due to microcrystals. These zones must have a new kind of amorphous arrangement which is induced by the flow mechanism.

Since the X-Rays scattering intensity was the same for any silica glass sample (deformed or not), we tried Raman microspectroscopy. Silica glass has given rise to many experimental studies and interpretations of the Raman spectra (especially of the two sharp lines D_1 and D_2). The intensity of these lines is known to be sensitive to thermal history /5/. Thus we have to be careful in our comparisons of the Spectra : a sample which has been deformed at a temperature T is compared with an undeformed sample, annealed in the deformation conditions (same temperature and same time of annealing). The thin sections examined previously are now put in a Raman microprobe. All Raman spectra are obtained in the back scattering configuration (laser power : 600 mW, wave length : 514,5 nm).

There was no difference in the Raman spectra between samples which have been deformed or annealed at a temperature above T_g but differences are obvious for below T_g experiments. Figure n° 1 shows the Raman Spectra in this case. The broad features are not modified but the intensity of the sharp lines has changed. Comparing figures n° 1a and 1b, we see that the deformation makes the line D_2 decrease. Any modification of the line D_1 is less evident.

When a deformed sample is annealed near the glass transition (figure n° 1c), the line D_2 comes back to its previous intensity. Provided that we are able to interpret the Raman Spectra, such experiments give an important information about the deformation mechanism. Owing to the convincing interpretation given by Galeener /3/, the decrease of the line D_2 can be interpreted as a modification of the number of threefold rings of tetrahedra. As there is about 1 % threefold rings in silica glass, it is a very slight modification of the ring statistic. Thus we can think to a new approach of atomic transport in glass based on this experimental result : under stresses SiO atoms are transferred from a ring to another one.

CONCLUDING REMARKS

These experiments have given some new structural knowledge on the deformation in silica glass.

Raman spectroscopy appears as a very keen technic in order to get some information concerning the ring statistic. We have yet some more investigations to do on deformed samples. Electron microscopy will be done on our sample to precise the local order of the birefringent zones.

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