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Induced anisotropy in oxide glasses

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Thermomechanically, optically, and chemically induced anisotropy in oxide glasses is reviewed. Optical anisotropy observed as birefringence and/or dichroism is correlated with form or distribution anisotropy in microstructure.

1. INTRODUCTION

Whereas the most common cause of optical anisotropy in glasses is stress, anisotropy of a different nature can be induced in glasses thermomechanically[1-3], optically[4], or chemically[5]. This area has fascinated a limited number of workers and, as a result, our understanding of the phenomena has come rather gradually.

Since in most cases the anisotropy in glasses has been observed as optical effects, we first briefly review selected behaviors of light in a medium[6], then discuss the reported processes and current interpretations of the observed anisotropy.

We are particularly interested in two types of interaction of light with medium: retardation and absorption. Retardation is the reduction of velocity of light when it travels through a medium. The propagation of light through an ordinary glass is slowed down to about two thirds of its velocity in vacuum. Absorption, on the other hand, is the reduction of energy while light travels through a medium. A portion of optical energy is usually converted to thermal energy, and the light intensity is reduced correspondingly. When light travels through a glass with anisotropic retardation characteristic, we observe birefringence. When light travels through a glass with anisotropic absorption characteristic, on the other hand, we observe dichroism.

Let's look at the behavior of light traveling through the anisotropic medium shown in Figure 1. This schematic figure is from Ernsberger's book[6] and shows a tetragonal crystal as an

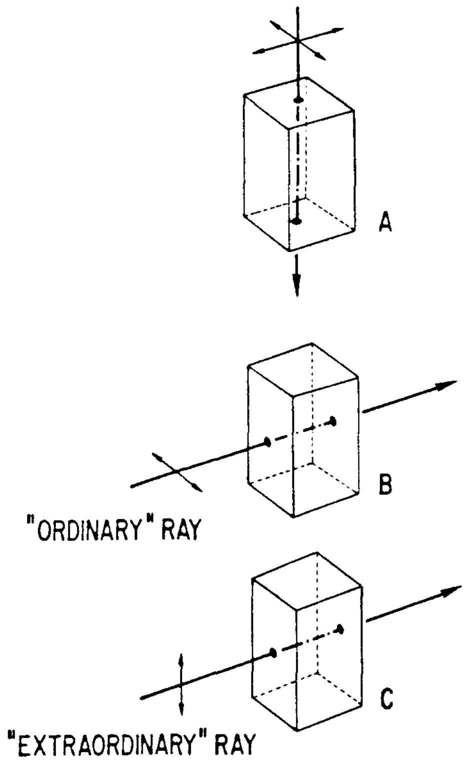


Figure 1. Interaction of light with a tetragonal crystal. (After Ernsberger[6])

example. When light propagates through the crystal parallel to the c-axis, as shown in A, its velocity in all planes of polarization is identical, and the light behaves as if it were propagating through an isotropic medium. The axis in such direction is called the optic axis. The c-axis of the tetragonal crystal here is parallel to the optic axis. When light propagates perpendicular to the optic axis, on the other hand, its velocity varies depending upon the plane of polarization. Light polarized in the plane perpendicular to the optic axis, as shown in Figure 1B, will have the same velocity as the light propagating parallel to the optic axis, and is called the "ordinary" ray. A light ray polarized in the plane parallel to the optic axis, as shown in Figure 1C, will have a different velocity from the ordinary ray since it sees a different electronic configuration in the medium. This is called the "extraordinary" ray.We will be referring to these terms, the optic axis, ordinary ray, and extraordinary ray, rather frequently in the later part of this paper.

The refractive index of a medium is a non dimensional number and is defined as the ratio of the velocity of light in vacuum to that in the medium. Thus, when light propagates in the direction perpendicular to the optic axis, the optically uniaxial material shown in Figure 1 exhibits two different refractive indices depending on the planes of polarization. The birefringence, B, in a material is defined as the difference in refractive index by the extraordinary ray, n_e, and refractive index by the ordinary ray, n_o, i.e.

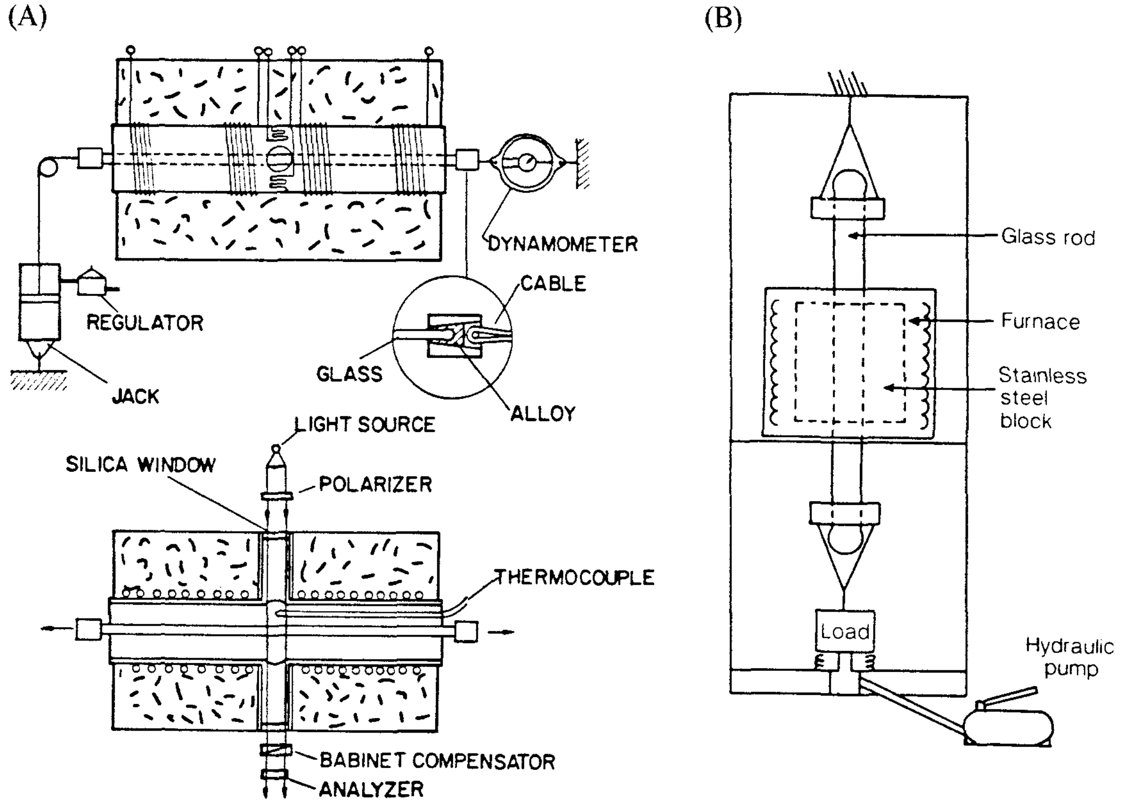


Figure 2. Laboratory setups for thermomechanical processing in the literature, (A) [14] (B) [15].

$$B = n_e - n_o \qquad (1)$$

Note that the birefringence may be positive or negative. The relative retardation, R, by this birefringence is

$$R = D(n_e - n_o) \qquad (2)$$

where D is the thickness of the material along the light path. Usually, the retardation per unit thickness, R/D (nm/cm), is used as a measure of the birefringence.

Experimental techniques and optical instruments used to measure the birefringence have been covered by numerous textbooks on stress optical effect[7-10].

With the above brief introduction, we now review the individual cases of induced anisotropy in glasses.

2. THERMOMECHANICALLY INDUCED ANISOTROPY

Thermomechanical processing is the most classic method to induce anisotropy in glasses[11-13]. Figure 2 shows simple laboratory setups for such processing. They consist of a furnace, a sample glass rod, and a pulling load. The setup in Figure 2A has polarizers attached to the furnace and allows measurement of any optical effects *in situ* at elevated temperature. With the one in Figure 2B, measurements are performed at room temperature after processing.

Sample glass rods are stretched in the viscoelastic temperature range, at a viscosity of 10^{10}-10^{11} poise and cooled under load to room temperature. Any glass can be made optically anisotropic by this processing, since the anelastic strain will be frozen-in[16], and display uniaxially positive birefringence corresponding to the tensile stress. The frozen-in strain is totally annealable by the ordinary procedure as reviewed previously[3], and will not be discussed any further in this paper. If the glass has second phase, on the other hand, the major part of the thermomechanically induced anisotropy persists even after ordinary annealing procedures, and is related to the anisotropic microstructure of the glass. We will focus on this kind of anisotropy in this paper.

2.1. Borosilicate glasses

Figure 3 shows the microstructure of a phase-separated borosilicate glass, Corning code 7070, which was heat treated at 600°C for 100 hours[3]. In this case, the spherical second phase is a low viscosity, borate-rich glass, and the matrix is silica-rich. When such phase-separated glass rods, after heat treatment for varied times at 595°C (prepulling heat

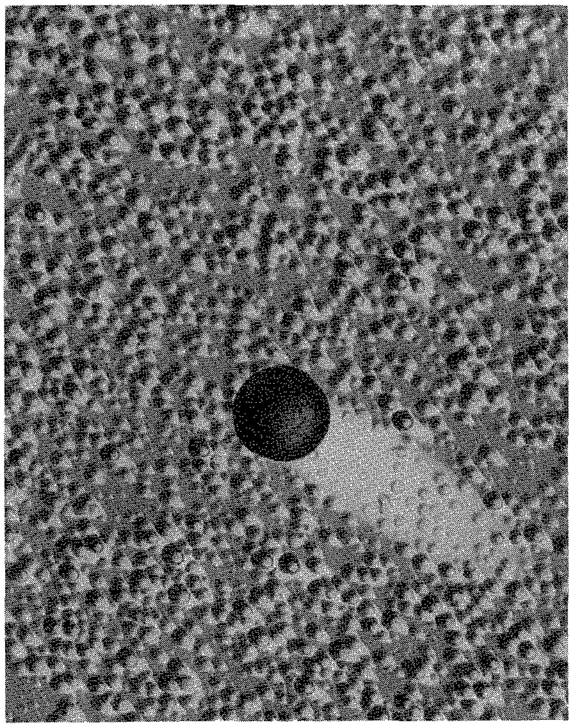


Figure 3. Microstructure of a phase-separated borosilicate glass (Corning code 7070). Glass was heat-treated at 600°C for 100 hr. Latex sphere shows 0.5 μm in diameter. (After Takamori and Tomozawa[3])

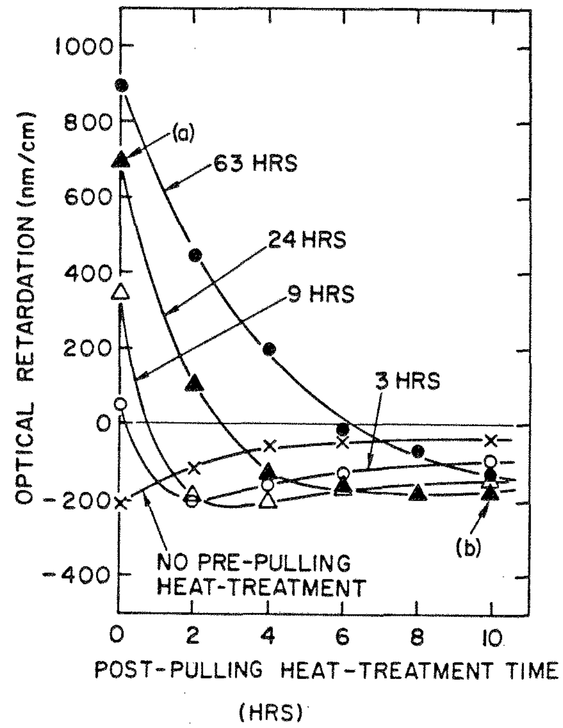


Figure 4. Birefringence observed as a function of the post-pulling heat-treatment time at 564°C. Times indicated on curves are the prepulling heat-treatment times at 595°C. (After Takamori and Tomozawa[17])

treatment), were stretched by twofold at this temperature and cooled under load, the samples showed strong positive birefringence even after annealing at its annealing point (496°C). The birefringence was uniform throughout the sample without any compensating negative birefringence as usually observed in the case of stress birefringence. During the additional heat treatment at 564°C (postpulling heat treatment), the positive birefringence relaxed quickly to negative values, and then asymptotically increased to zero. These observations are summarized in Figure 4[17]

The typical microstructure of samples exhibiting positive and negative birefringence is shown in Figure 5, (a) and (b), respectively[3]. In Figure 5(a), we can see that the second phase particles are elongated and aligned in the direction of stretching, thus indicating that the observed positive birefringence is caused by the aligned prolate spheroids. This is a well known effect called form birefringence, which has been studied by several other investigators[18-26]. The microstructure of the sample with negative birefringence, on the other hand, contains spherical second phase as seen in Figure 5(b). Then, the question which remains is, "what is the cause of the negative birefringence?".

Figure 6 shows the possible sequence of the events[17]. First, the isotropically dispersed spherical second phase particles in Figure 6(a) are stretched to prolate spheroids shown in Figure 6(b), which results in positive form birefringence, and upon subsequent heat treatment the prolate spheroids return to their initial spherical shape but stay at the same position, resulting in the anisotropy in distribution, which would cause the negative birefringence observed in Figure 4. The present authors proposed to call this type of birefringence *distribution birefringence*[17].

Seward has studied similar anisotropy using a lead borate glass[27]. The sample glass in this case was stretched over a much lower viscosity range, such as 10^7 poise, at high speed. The second phase was elongated to an aspect ratio as much as 100:1, as shown in Figure 7(a). Then, subsequent heat treatment caused the local breaking up of the second phase to spheroidize as shown in Figure 7(b). Unfortunately, any change in optical effect has not been pursued in this case, however[28].

As described above, the form birefringence has been studied by several investigators in different areas. The anisotropy in distribution, however, has rarely been singled out for analysis. Also, the above observations on a phase-separated borosilicate glass is a rare example where one can clearly separate the

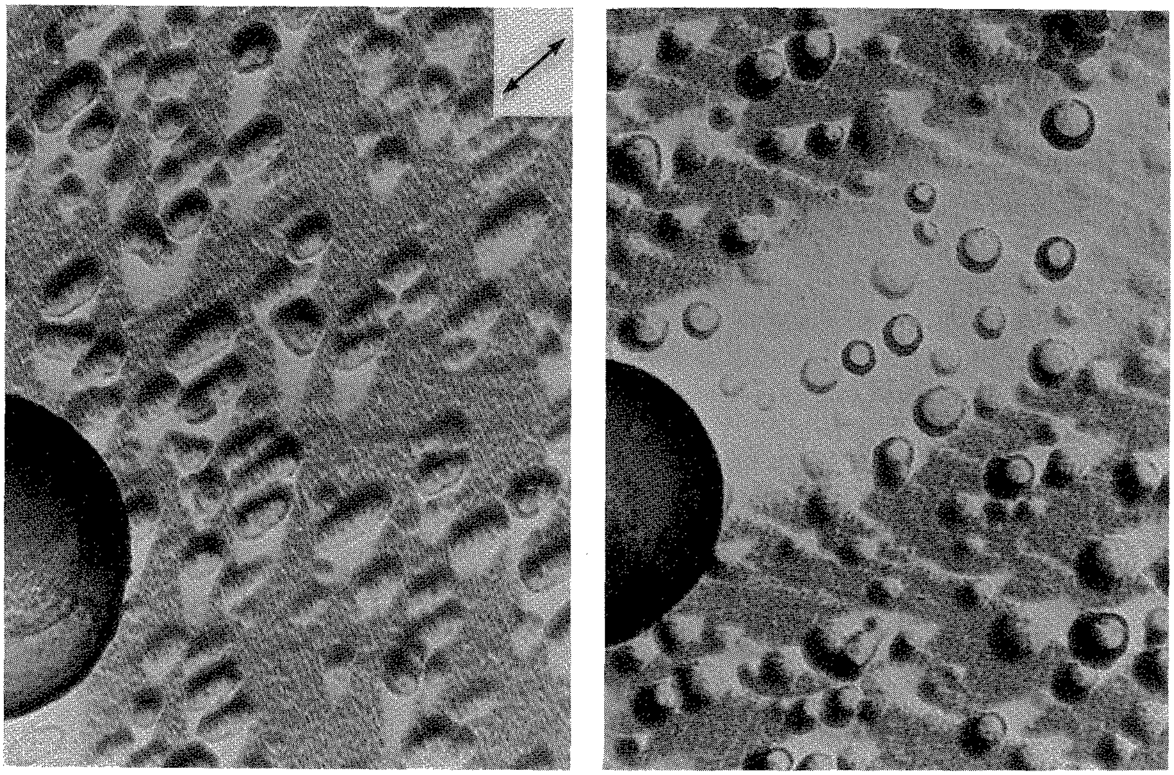


Figure 5. Microstructure of the stretched borosilicate glass. The glass was subjected to 100 hr of prepulling heat treatment at 595°C and stretched. (left) before and (right) after the postpulling heat treatment at 600°C for 16 hr. The arrow indicates the stretching direction. Latex sphere shows 0.5 μm in diameter. (After Takamori and Tomozawa[3])

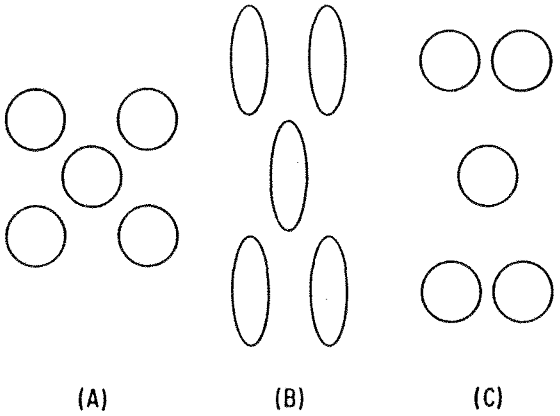


Figure 6. Schematic representation of microstructures. (a) After the prepulling heat treatment, the sample is phase separated but still isotropic. (b) After pulling, the sample is anisotropic with a uniaxially elongated second phase and gives positive birefringence. (c) After the postpulling heat treatment, the sample is still anisotropic due to the anisotropic distribution of the spherical second phase and gives negative birefringence. (After Takamori and Tomozawa[17])

distribution effect from the form effect. In many other cases where form and distribution effects coexist, the form effect dominates and, therefore, it is difficult to study the effect of distribution

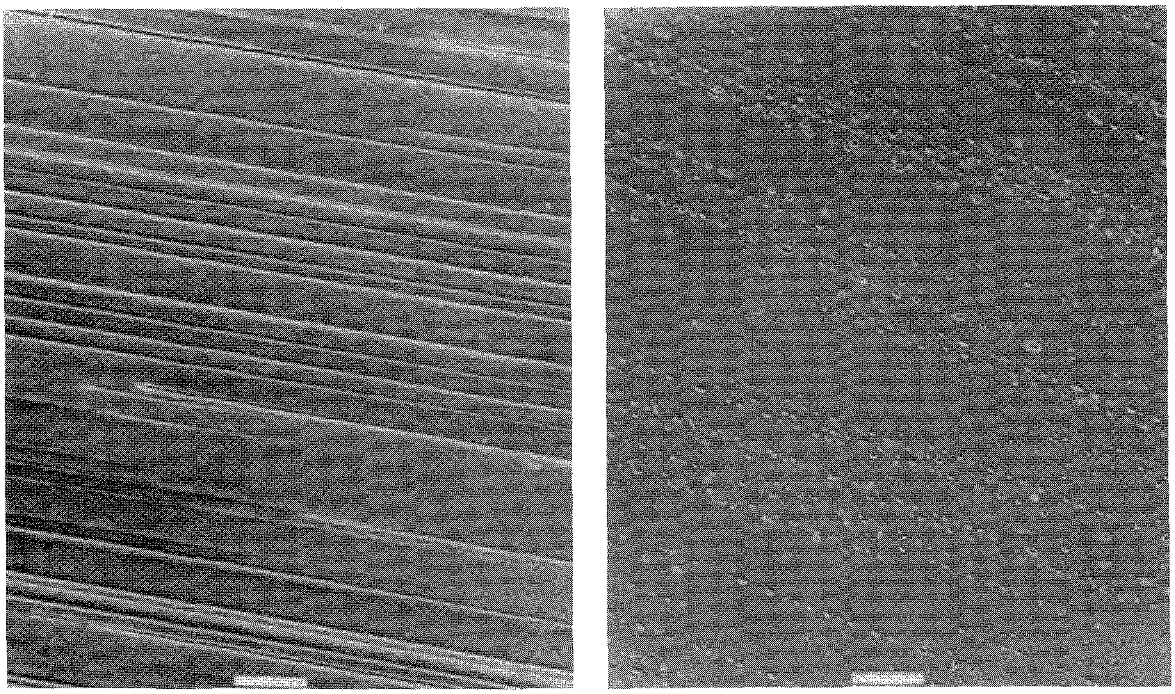
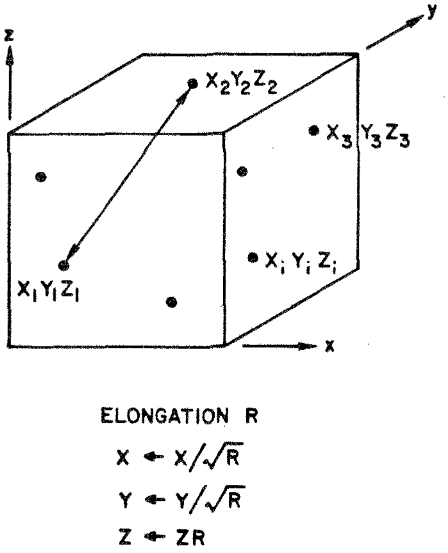


Figure 7. Microstructure of (left) a phase-separated and stretched lead borate glass (PbO 40 wt%, B_2O_3 60 wt%), and (right) after postpulling heat treatment at 490°C for 40 min. (After Seward[27])

Figure 8. Computer simulation of distribution anisotropy. Initially cubic space containing randomly distributed spherical particles of finite size is stretched in z-direction keeping its volume constant.



anisotropy. Earlier, we attempted to interpret the observation on the basis of Sillars' equation, and obtained positive birefringence for form anisotropy, negative birefringence for distribution anisotropy, and as sum of these, positive birefringence[3]. At least qualitatively, the results were consistent with the observations. Recently, Nan and Smith[29] revised our earlier treatment on the basis of light scattering by particles and effective-medium theory. They showed that the total birefringence increases with increasing size of the second phase and size distribution variance, as well as volume fraction and the refractive index difference between matrix and the second phase.

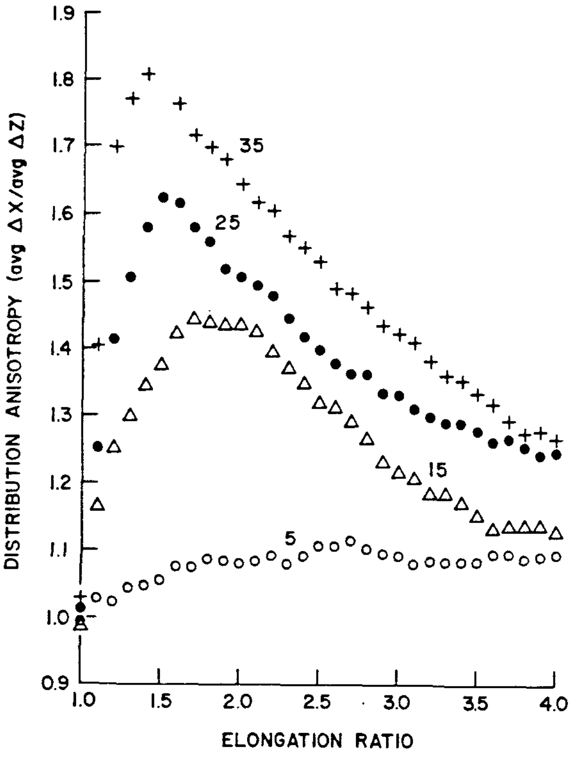


Figure 9. Distribution anisotropy, defined as the ratio of average x and z components of nearest neighbor distance, as a function of elongation ratio. (After Takamori and Tomozawa[30])

In order to confirm the distribution anisotropy in the random system, we also attempted a simple computer simulation; its schematic representation is shown in Figure 8[30]. Using a random number generator, we assigned coordinates to points in finite space, and elongated the space R times keeping the volume constant and assuming the points have finite size. Figure 9 summarizes the results which indicate the existence of a maximum anisotropy, with its position shifting to smaller elongation with increasing population of points. The data in Figure 9 were used for theoretical calculations by Nan and Smith[29]. The results of the calculations by Nan and Smith, which are plotted in Figure 10(b), are consistent with the experimental results by Thomas[14] on the negative birefringence of stretched samples in Figure 10(a).

2.2. Silver-containing glasses

When small metallic particles are dispersed in a glass, they absorb and scatter light. If the particles are small enough, the glass remains transparent, but colored. By stretching such a glass, one can obtain optically anisotropic glasses. The anisotropy observed in this case is not birefringence but dichroism[31]. The second phase here is metallic silver, and the amount added is at most 0.5 weight %, but the effect is significant. Metallic silver precipitation is obtained by heat treating the silver-ion-containing glass before stretching. Figure 11 shows an elongated silver metal particle obtained by Stookey and Araujo[31]. They stretched a heat-treated glass from 50 to 500 times the original length at a viscosity of about 10^7 poise, but the aspect ratio of 3:1 as seen here seems to be just about the highest obtainable. They observed strong absorption of extraordinary ray by such a stretched glass, and reported that the color changed continuously from orange to blue as the elongation increased. Figure 12 shows more recent data by Seward[32], in which absorption of extraordinary ray (indicated by parallel marks) increases with increasing elongation of silver particles in the glass. At the same time the wavelength of absorption increases with increasing elongation of silver particles. Absorption of ordinary ray (indicated by perpendicular marks) by the same glasses does not change much both in strength and wavelength. Because of the change in absorption wavelength of extraordinary ray, the resultant sample colors changed as indicated on the peaks of the absorption curves.

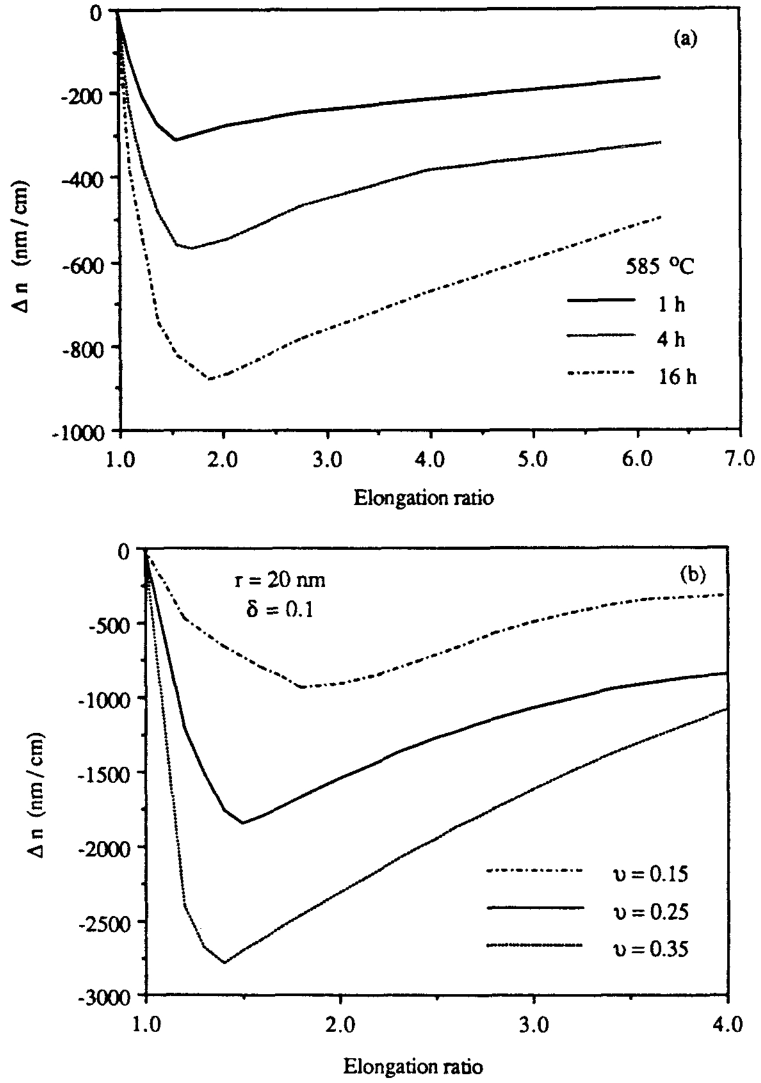


Figure 10. Microstructural birefringence as a function of elongation ratio. (a) The experimental results of stretched commercial borosilicate glass after postpulling heat treatment under different conditions (replotted data by Thomas[14]). (b) The theoretical distribution birefringence. (After Nan and Smith[29])

2.3. Photochromic glasses

The second phase present in photochromic glasses is silver halides which are precipitated by the heat treatment before stretching. Figure 13 shows the elongated silver halide particles after stretching at three levels of pulling stress, stress increasing from top to bottom figure[33]. These stretched glasses reveal dichroism at darkened state by the metallic silver precipitation, and birefringence at clear state. In spite of the much lower volume fraction (<1 %) of the silver halide particles in these glasses compared to that of the second phase of the phase-separated borosilicate glasses,

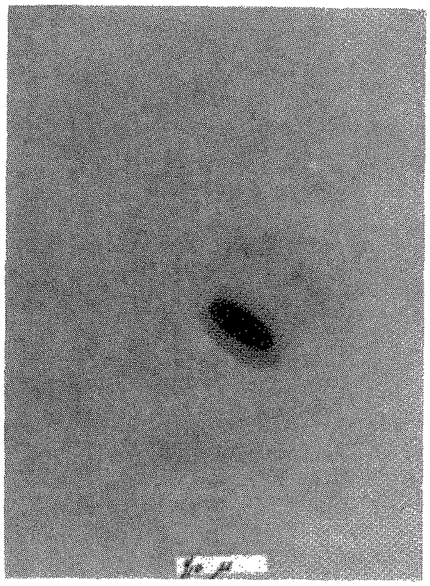


Figure 11. Elongated silver metal particle in glass (After Stookey and Araujo[31]

Borrelli[33] reports birefringence of a photochromic glass showing as high as 2900 nm/cm at 633 nm wavelength. This result can be attributed to the high refractive index of silver halides (>2)[34] and the high aspect ratio (>30:1) of the second phase particles attainable[35] in these glasses. On the other hand, if an attempt is made to increase the aspect ratio of the second phase particles in borosilicate glasses by elevating the stretching temperature so that the glass can be stretched in lower viscosity range at high speed, it would automatically approach to the immiscibility boundary[36] such that the composition difference between matrix and the second phase decreases, and so does the refractive index difference.

2.4. Polarcor^{TM}

Development of Polarcor^{TM} is apparently the result of the extension of the study of elongated photochromic glasses at Corning[37]. It seems to be the only product commercially available in this area of study.

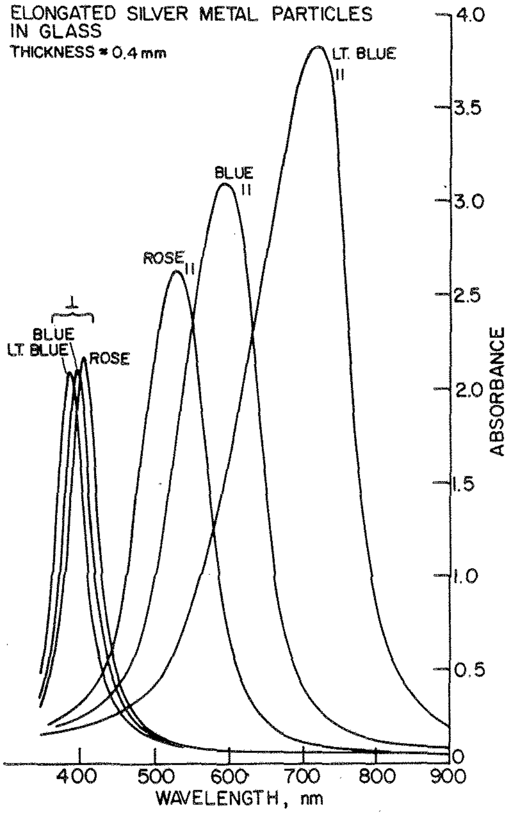


Figure 12. Different polarization behavior of stretched glass resulted from different degrees of silver particle elongation. (After Seward[32])

The silver halide-containing glass is first heat-treated rather extensively in this case compared to the normal heat treatment for photochromic glasses to attain larger silver halide precipitates[38]. The glass is then stretched thermomechanically, producing a strong birefringence as discussed in the previous section. At this stage, the glass is not dichroic. The stretched glass is now subjected to additional heat treatment in hydrogen atmosphere at temperatures between about 200 °C and its annealing point. This treatment chemically reduces the silver halide to silver metal which is confined within the elongated

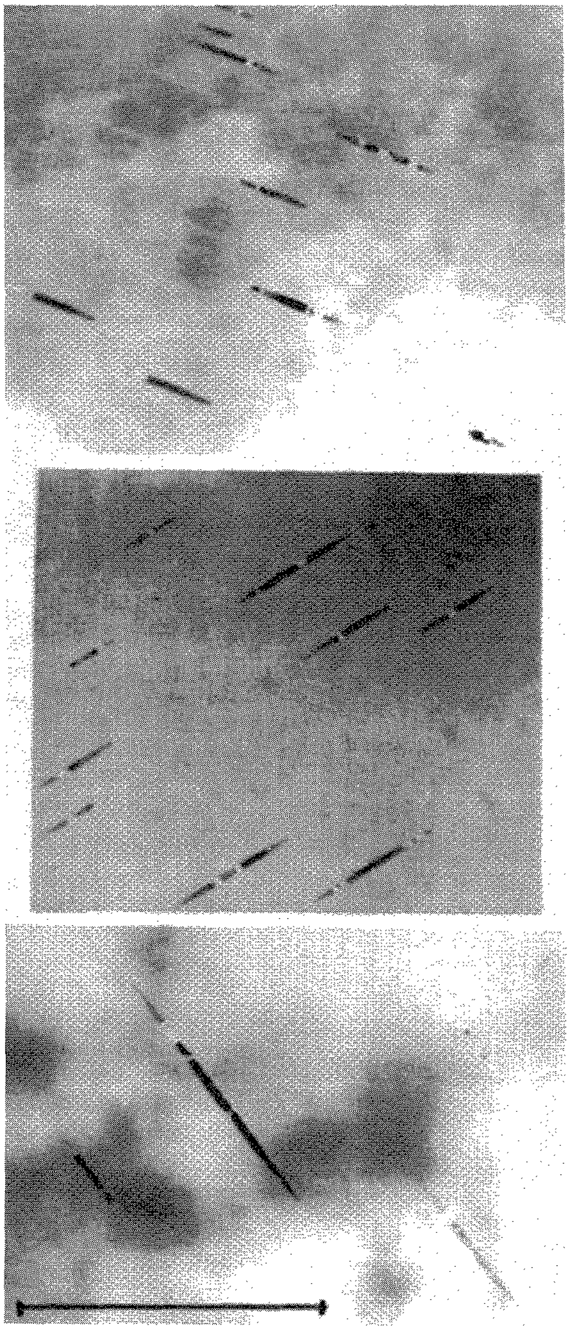


Figure 13. Electron micrographs of silver halide particles in a photochromic glass after stretching at three levels of pulling stress, stress increasing top to bottom. Bar at the bottom indicates 0.5 μm (After Borrelli[33])

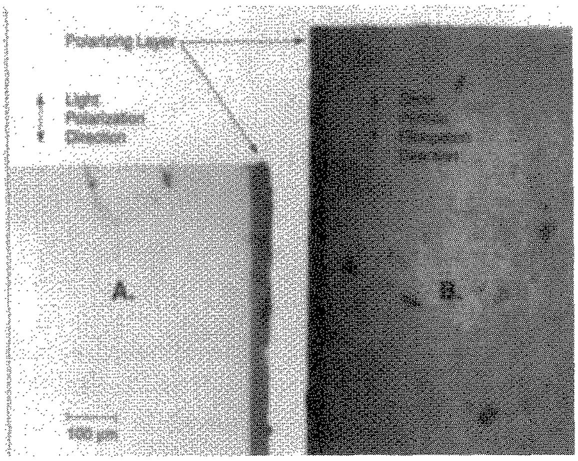


Figure 14. Polarcor^{TM} polarizers viewed on edge in polarized light. (After Taylor[37])

envelope of the original silver halide particle. By this thermochemical treatment, a 20-50 μm thick surface layer on both sides of the glass become dichroic with strong absorption of extraordinary ray in the near IR region[32]. With this process, it became possible to produce silver particles having higher aspect ratio than 3:1 in stretched glasses.

Figure 14 shows a Polarcor viewed on edge in polarized light[37]. The surface layer of 30 μm contains long silver particles, reduced from elongated silver halide particles, which selectively absorb the extraordinary ray. In this figure, the polarization direction of light is in the plane of the paper as indicated. In sample A, the elongated silver particles are aligned perpendicular to the plane of the paper, and thus the light polarized in the plane of the paper is the ordinary ray for the sample. No extinction occurs in this case except only partial absorption. In sample B, on the other hand, the silver particles are aligned parallel to the plane of the paper as indicated, and thus the light polarized in the plane of the paper is the extraordinary ray for the sample. A significant extinction occurs in this case.

Figure 15 is an example of transmittance curves[37] of a Polarcor (a) for ordinary ray and (b) for extraordinary ray. Note that the curve for the latter is in log scale. It is reported that the polarizer with contrast ratio of greater than 10,000:1 is available. Similar to Figure 12, varying aspect ratio of the silver particles should give wavelength selection for specific applications.

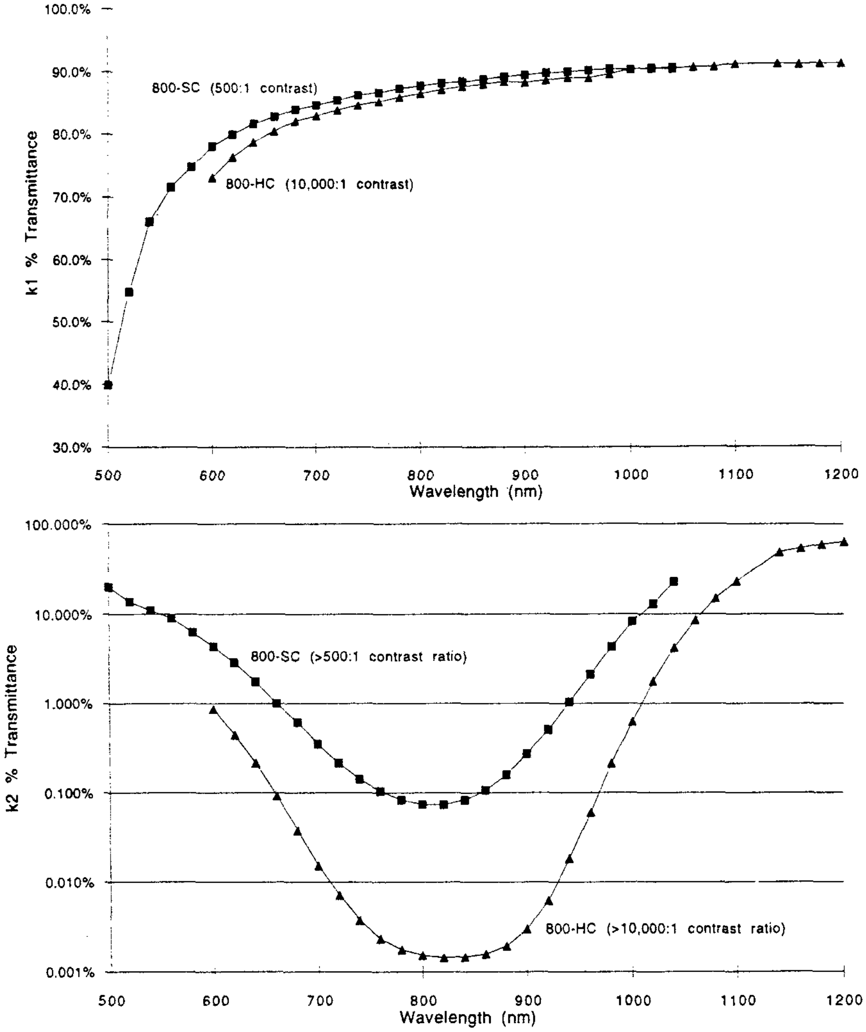


Figure 15. Transmittance curves of Polarcor^{TM} polarizers of high and standard contrast ratio designs (a) by ordinary ray, and (b) by extraordinary ray. (After Taylor[37])

3. OPTICALLY INDUCED ANISOTROPY

It is well known that photochromic glasses darken when exposed to radiation in the near UV and blue spectral regions, and fade (i.e. clear) when irradiation ceases. When they are exposed to intense visible radiation, the rate of fading is often enhanced by an effect called optical bleaching[39].

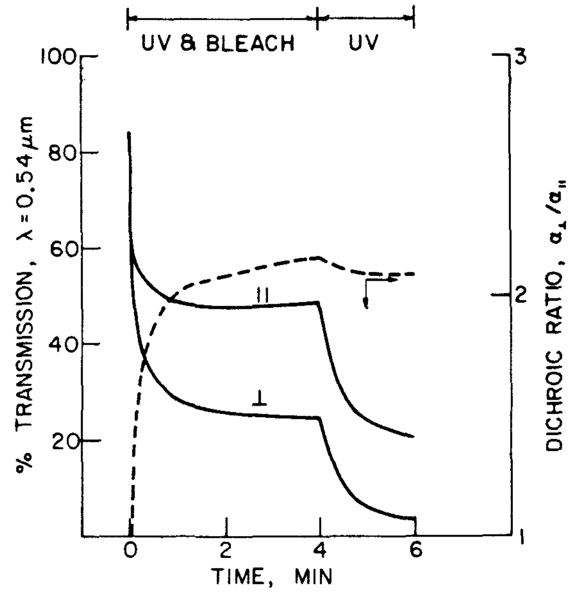
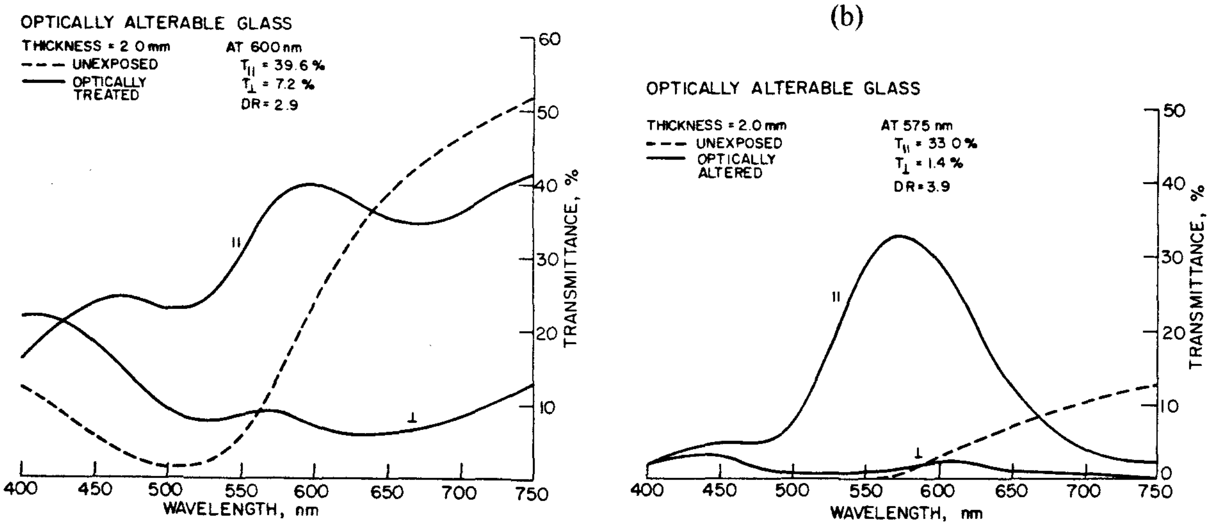


Figure 16. Optically induced dichroism in a silver-halide-type photochromic glass. Solid curves: glass transmittances. Dashed curve: dichroic ratio calculated assuming 8% transmittance loss due to surface reflections. Sample thickness is 2.0 mm. (After Borrelli and Seward[4])

When Borrelli and Seward[4] attempted such optical bleaching using a polarized laser light, they found that the sample became dichroic revealing a greater transmission of the light polarized in the direction parallel to the polarization direction of the bleaching light than that in the direction perpendicular to it. Figure 16 is one of their data on the transmission as a function of time when the sample glass is exposed to the darkening UV and the bleaching light simultaneously. In this figure, the initial decrease of the transmission is caused by the UV and the splitting into two curves is caused by the polarized bleaching light. The transmission of the light polarized in the direction parallel to the polarization of the bleaching light (indicated by parallel mark) is almost twice of that in the direction perpendicular to it (indicated by perpendicular mark) in this case.

In addition to the dichroism, birefringence was also observed. The optic axis of the birefringence was determined to coincide with the polarization direction of the bleaching light[40]. That is, the light ray of measurement polarized in the polarization direction of the


(a)

(b)

Figure 17. Transmittance spectra of optically alterable glass in two cases, (dashed curves) before and (solid curves) after optical treatment. (After Seward[32])

bleaching light is the extraordinary ray. Since the light ray in this direction was found to be faster(lower index), the measured birefringence in the range of 100-1000 nm/cm at 850 nm[40] must be negative in sign, according to the definition by Equation (1). The negative sign here indicates that the observed form birefringence is caused by oblate, or disk-like, microstructure, which is consistent with the model deduced from other experiments[40, 41].

Further, similar effects have been shown on non-photochromic glasses with high silver content[32]. Silver was added in the form of nitrates and chlorides in the batches for melting. The glasses obtained were highly colored in red-brown to almost opaque because of the high silver content. Examples of spectra for these initial glasses are shown by dashed curves in Figure 17. When these glasses were irradiated with strong polarized radiation in visible to near IR range, they became dichroic as shown by solid curves in Figure 17. The transmission was significantly greater for the light ray polarized in parallel to the irradiated polarized light (the extraordinary ray, indicated by parallel mark) than the light ray polarized in perpendicular to it (the ordinary ray, indicated by perpendicular mark). Also the dichroism was greatest at the wavelength of the treating radiation, which is 600 nm for the sample in Figure 17(a), and 575 nm for that in Figure 17(b).

A unique feature of the optically induced dichroism here is that it can be altered repeatedly both in wavelength and in direction of polarization by the subsequent irradiation.

Optical anisotropy has also been reported when Fe^{3+}-containing silicate and phosphate glasses were irradiated by γ-ray and then subjected to optical bleaching by UV lasers. The induced dichroism has been attributed to the anisotropy in hole color centers[42, 43].

4. CHEMICALLY INDUCED ANISOTROPY

When borosilicate glasses of certain range of compositions are phase separated into silica-rich phase and borate-rich phase, each of these phases is interconnected, and the borate-rich phase can be leached out by acid to produce a silica-rich microporous glass. When this microporous glass is soaked in a liquid, it is often found to be birefringent as shown in Figure 18(B)[3]. The magnitude of such birefringence changes continuously with the refractive index of the penetrating liquid, and when the refractive index of the liquid is close to that of silica glass, 1.46, the birefringence disappears as shown in Figure 18(A).

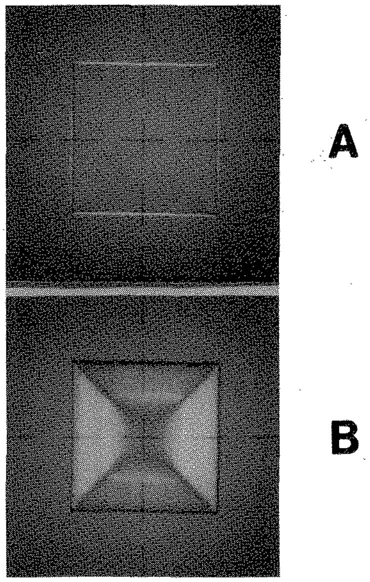


Figure 18. Polariscopic pictures of a microporous glass, immersed in a liquid with a refractive index of (A) 1.46 and (B) 1.32. (After Takamori and Tomozawa[3])

The optic axis for such a microporous glass has been determined as follows[5]. First, the microporous glass rod was prepared by phase separation and leaching. When it was sliced in the direction parallel to the rod axis and soaked

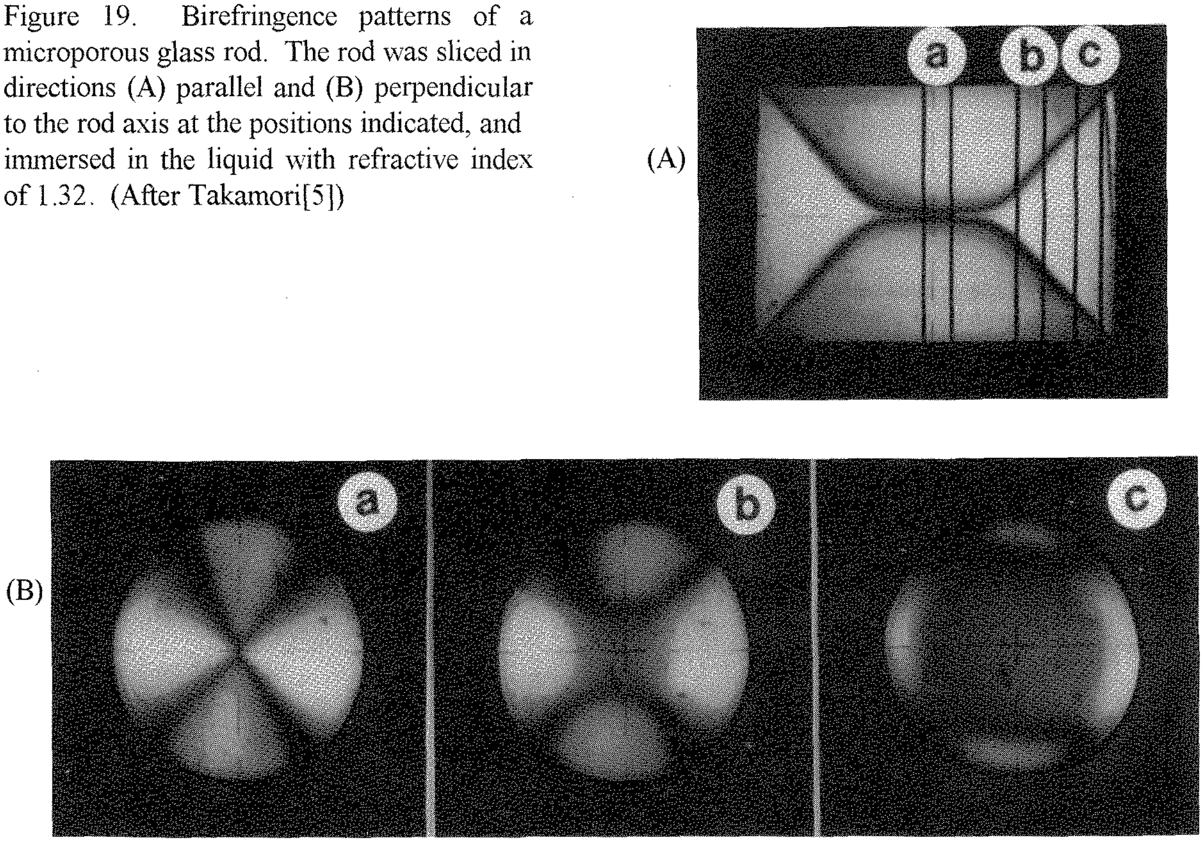


Figure 19. Birefringence patterns of a microporous glass rod. The rod was sliced in directions (A) parallel and (B) perpendicular to the rod axis at the positions indicated, and immersed in the liquid with refractive index of 1.32. (After Takamori[5])

in a liquid with a refractive index of 1.32 (close to that of water), its birefringence pattern viewed perpendicular to the rod axis was as shown in Figure 19(A). The same rod was sliced in the direction perpendicular to the rod axis at the positions (a), (b), and (c), and was soaked in the same liquid to observe the birefringence pattern in the direction parallel to the rod axis. As shown in Figure 19(B), the central areas of (b) and (c) were not birefringent, indicating that the conical portion of the rod in Figure 19(A) viewed in the direction of the rod axis does not exhibit birefringence. That is, the light behaves as if it were propagating through the isotropic medium in this direction. Therefore, this is the optic axis for the conical portion of the rod. This conical portion corresponds to the region where the leaching proceeded in the direction parallel to the rod axis. Therefore, the optic axis of the anisotropy in this case is parallel to the direction of material transport during leaching. On this basis, the sign of birefringence is easily determined to be negative.

By soaking a slice of the microporous glass in a 0.5N KOH solution for several hours, the birefringence pattern previously observed can be erased entirely. SEM micrographs in Figure 20, (A) before and (B) after the soaking in KOH, clearly show that the gel-like structure, which is known to exist as the result of dissolution and precipitation during the leaching procedure, has been dissolved away by this treatment. This confirms that the anisotropy in the microporous glass is developed during the leaching process, and, therefore, it can be dependent on the leaching condition. For example, Figure 21 shows how the birefringence

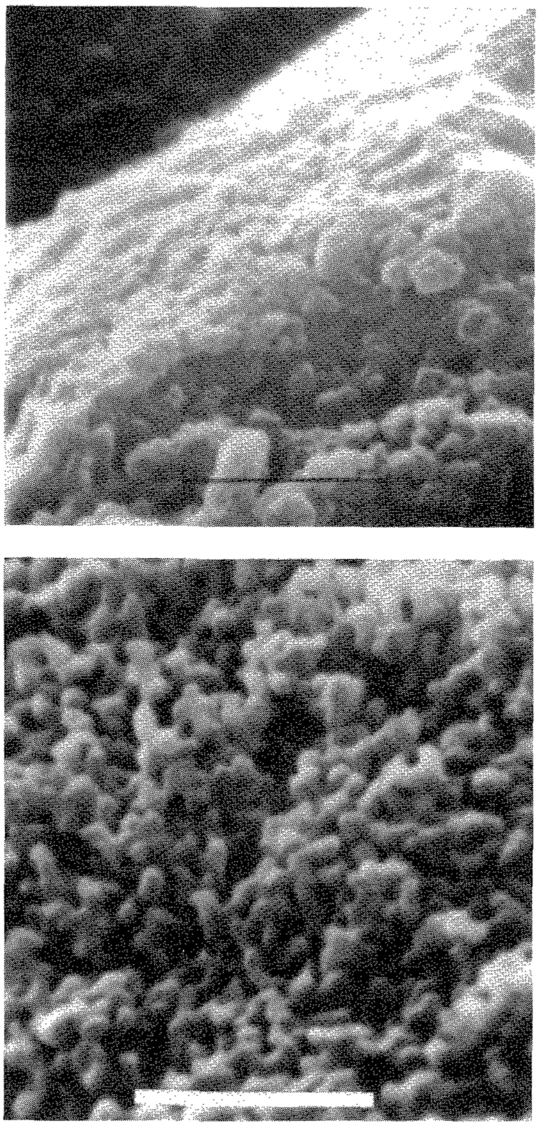


Figure 20. SEM micrographs of fractured surfaces of the microporous glasses (A) before and (B) after treatment with 0.5 *N* KOH solution. Bar = 1 μm. (After Takamori[5])

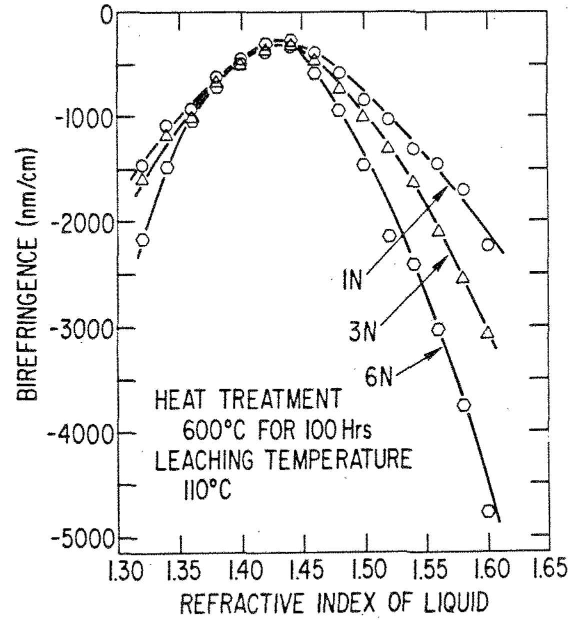


Figure 21. Effect of leaching acid concentration on the birefringence observed in microporous glasses. (After Takamori[5])

of a microporous glass may be altered by the leaching acid concentration. Further studies on the leaching condition dependency of birefringence have been reported recently[44, 45]. However, the effect of the initial glass composition, which should affect the leaching condition, has rarely been discussed[5].

5. CONCLUDING REMARKS

We have reviewed several cases of induced anisotropy in oxide glasses mainly from a phenomenological point of view. Some of the topics which we covered in the previous review but did not in this paper are anisotropy by frozen-in strain and that by crystallizing glasses. The main reason for this omission is that we did not come across noteworthy works on these topics in the past decade.

As we mentioned at the outset, the progress in this area has been rather slow, and there are many interesting questions which remain to be answered in future work. For example, (1) in the study of thermomechanically induced anisotropy, what if the borosilicate glasses contain elements which absorb light?

(2)Apparently, distribution anisotropy should invite more theoretical elaboration. (3) Also, its contribution to dichroism in any case has not been discussed. (4) More work on optically induced anisotropy is certainly desired. (5) Our current knowledge on the structure of gel in microporous glasses, which is responsible for the observed birefringence, is no more than a speculation.

Anisotropy in other than optical properties are also expected in the structurally anisotropic glasses. In fact, anisotropy in dielectric properties has been observed for the borosilicate glasses discussed in this paper[46].

Finally, we hope more workers will find interest in this fascinating area in the future.

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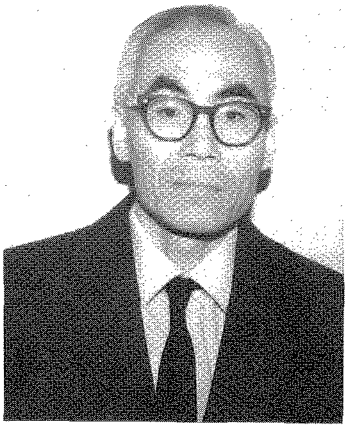

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