THE WARPING AND CRACKING OF PLEXIGLAS™ SPECIMEN CONTAINERS

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Abstract.—In the 1960s Plexiglas™ specimen containers were introduced in the anatomical collection of the Leiden Medical Faculty and in time replaced the cast rectangular glass jars. It was assumed that the Plexiglas™ jars would be less fragile and therefore better suited for use in medical teaching. Recently, we noticed that Plexiglas™ jars, after being filled and sealed, gradually warp to the inside and finally crack at the glued joints. We studied three experimental models to determine that the causes for the warping and cracking of Plexiglas™ jars are the result of the absorption of water by the Plexiglas™ and its slow diffusion through the acrylic.

For more than 300 yr, fluid-preserved specimens have been an important tool in medical teaching. For demonstration purposes, it is essential that the specimens are stored in clear transparent containers. Until the 1930s, glass was the only suitable material. Initially, only cylindrical shaped jars were used, but by the end of the 19th century, rectangular-shaped jars were also available. The great advantage of these jars is their lack of visual distortion. However, because of the fragility of glass, the jars were kept in cabinets and rarely allowed in the hands of students.

In the early 1930s, polymethylmetacrylate (PMMA) was introduced under the names Perspex™ and Plexiglas™. This new synthetic material was, like glass, clear, transparent, and highly water resistant. The cast sheet material can be cut to a preferred size and shaped by thermoforming. Sheets can be glued together with chloroform or a polymer/monomer slurry (PMMA/MMA).

In the Leiden Anatomy Museum, Plexiglas™ jars were introduced in the collections in the 1960s. They had a great advantage for medical teaching compared to glass containers. Because of its flexibility, PMMA seemed to be less vulnerable to cracking and therefore safer to handle. We discovered that, in time, there were also disadvantages in the use of acrylic jars (van Dam 2000). It appeared that the Plexiglas™ jars, which were filled with either 10% formalin (3.6% formaldehyde in water) or 75% glycerin (64% glycerol in water), gradually warped inward after being filled and sealed (Fig. 1). Although for periods of 10 yr or more the containers showed no visual decline in fluid level, the level dropped significantly (5–10%) when the container was aerated. The glued joints showed little cracks from the outside inward (Fig. 2). These observations indicated that the sides of the jars had been under considerable stress. Consequently, upon inspection, several jars were found leaking and a few jars spontaneously burst at the joints. Apparently, when the jars are aerated, the observed fluid loss is not compensated for by air intake, and consequently a negative pressure is created inside the jar (van Dam 2000). This causes the jar to warp to the inside and the glued connections to

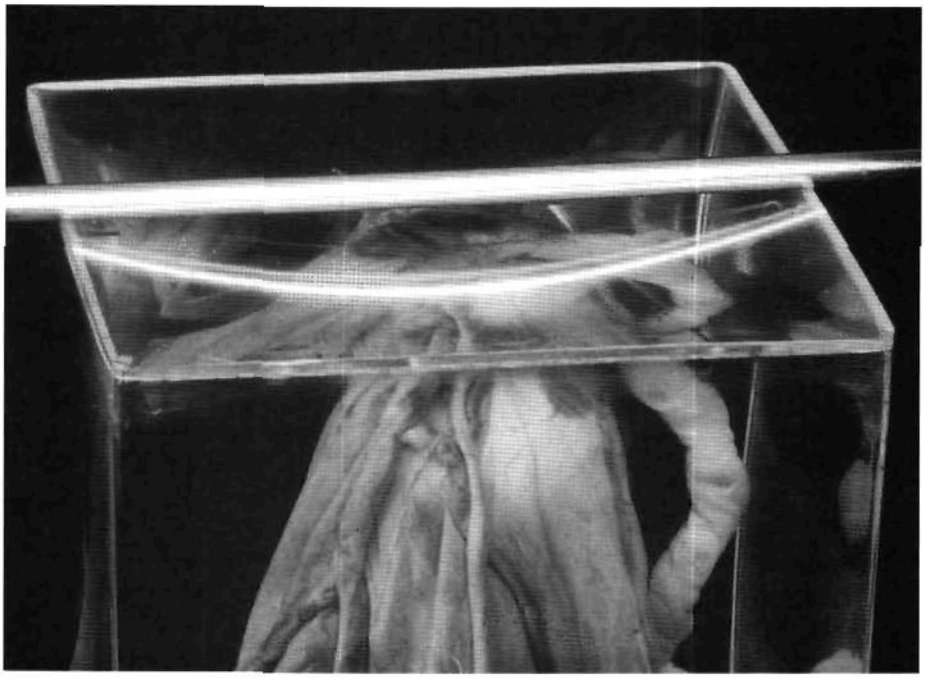

Figure 1. Warped Plexiglas® jar. The reflection of the metal rod shows the extent of warping of the upper side.

Figure 2. Partially split seam of a Plexiglas® jar.

weaken from stress cracks. In this fragile condition these specimen jars are hardly suitable for medical teaching.

At present, specimens in leaky Plexiglas™ containers, and newly prepared specimens, are placed in cast rectangular glass jars. However, these are difficult to find now, and are available only in a few sizes. For these reasons, there is an urgent need to determine the causes for the warping and cracking of Plexiglas™ to find solutions to these problems. In this paper, several experimental models were used to study the mechanism of the warping and cracking of fluid filled, sealed Plexiglas™ containers. The causes and possible solutions for these problems are discussed in the context of the experimental results.

Materials and Methods

Three experiments were carried out to determine whether the observed fluid loss, negative pressure, and warping of the jars could be related to the permeability and absorption properties of Plexiglas™.

Absorption experiment.—The purpose of this experiment was to determine which components of the fluid are absorbed most readily by the Plexiglas™. Small Plexiglas™ sheets (25 × 30 × 6 mm) were placed in demineralized water, glycerol, and formaldehyde solutions for 50 days. Immediately before and after the experiment the sheets were weighed. The next two experiments were based on the outcome of the absorption experiment.

Pressure experiment.—The purpose of this experiment was to determine if, in a hermetically sealed Plexiglas™ jar, fluid loss by diffusion could be related to the observed negative pressure in the Plexiglas™ jars in the museum. An 8 mm thick cylindrical Plexiglas™ container with a volume of 560 ml was totally filled with demineralized water, excluding as much air as possible. Pressure and temperature inside the jar were monitored by sensors and recorded by means of a computer, so that the jar could be disconnected during the experiment for weighing (Fig. 3).

Warping experiment.—The purpose of this experiment was to determine if there would be a difference in the extent of warping in closed and open (vented) systems. Two cylindrical glass containers, each with a diameter of 35 cm and volume of 10 L were filled with demineralized water and closed with a 2-mm thick Plexiglas™ lid with a 6-mm drilled hole approximately 2 cm away from the outer edge. The lids were sealed with bitumen (Shell Tixophalte®). Both jars were topped up with water to a level of 1.5 cm below the rim, and the holes in the lids were left open. One day later, one of the jars was totally filled, including some small air pockets underneath the lid. The hole in the lid was closed with a small sheet of 2-mm thick Plexiglas™, which was sealed with Acrifix 190 to make the closed system. The vertical displacement of the center of the lids was monitored by a micrometer with its sensor tip placed in the center, on top of the lid. A temperature probe was placed close to the jars to record ambient temperature (Fig. 4).

Results

Absorption experiment.—Table 1 shows the weight change of the Plexiglas™ samples after having been soaked in water and different concentrations of glycerin and formalin. The results of this experiment show that the higher the water content

Figure 3. Experimental setup to measure the pressure drop inside a water-filled, sealed Plexiglas® container.

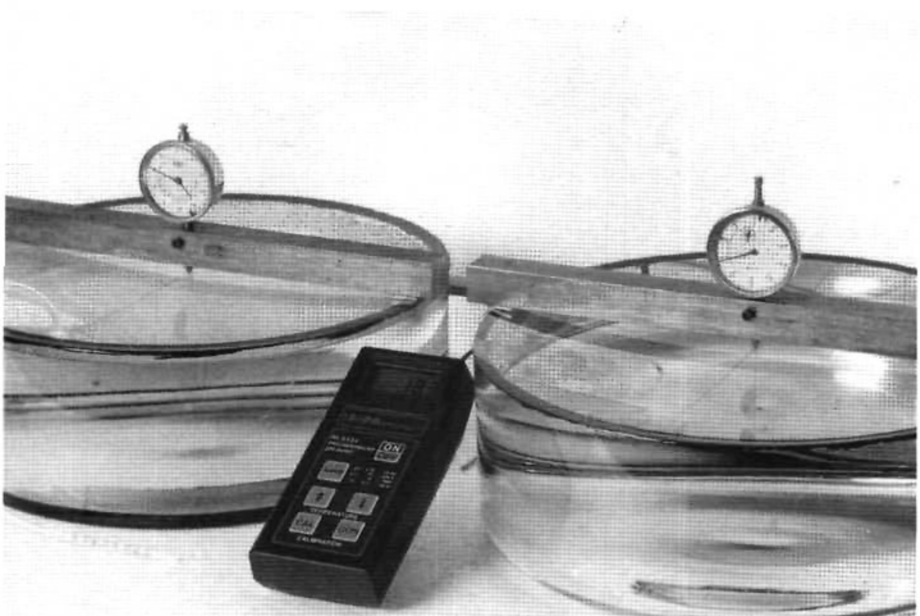

Figure 4. Experimental setup to measure the inside warping of Plexiglas® in open and closed containers filled with water.

Table 1. Weight increase of Plexiglas® samples after immersion in water, formalin, and glycerin for a period of 50 days.

Fluid	Concentration (% w/w)	Weight increase (%)
Formaldehyde in water	3.3	0.707
	9	0.644
	18	0.558
	Concentration (% v/v)	
Glycerol in water	20	0.662
	30	0.539
	45	0.428
	67	0.204
	100	−0.394
Water	100	0.750

in the mixture, the greater the weight increase of the sample. The greatest increase can be seen in the sample soaked in pure water. A decrease in weight was recorded for the sample soaked in 100% glycerol.

Pressure experiment.—The pressure/time graph for the water-filled, sealed Plexiglas® cylinder (Fig. 5) shows the decrease in internal pressure during a time period of approximately 200 days measured at an ambient temperature of 22.0 ± 0.5°C. The pressure readings on the internal probe dropped in the first month to −0.1 atm (−103 g/cm^2). From this point, it took another 4 mo to drop to a reading of −0.2 atm (−207 g/cm^2). The graph shows a linear regressive trend in the first 30 days followed by a less regressive trend from day 50 onward. The weight readings recorded a loss of 1.5 g at day 110 and 1.7 g at day 204.

Warping experiment.—The graph for the warping of the Plexiglas® lids (Fig.

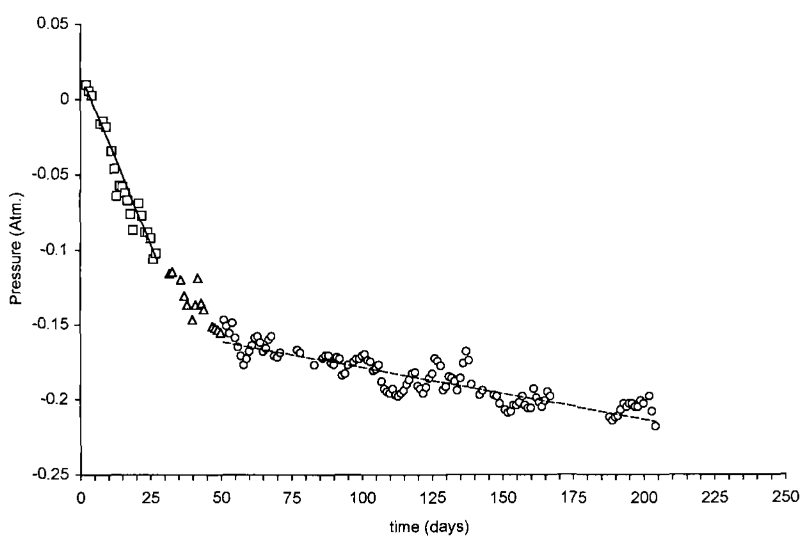

Figure 5. Pressure/time graph for the water-filled, sealed Plexiglas® vessel.

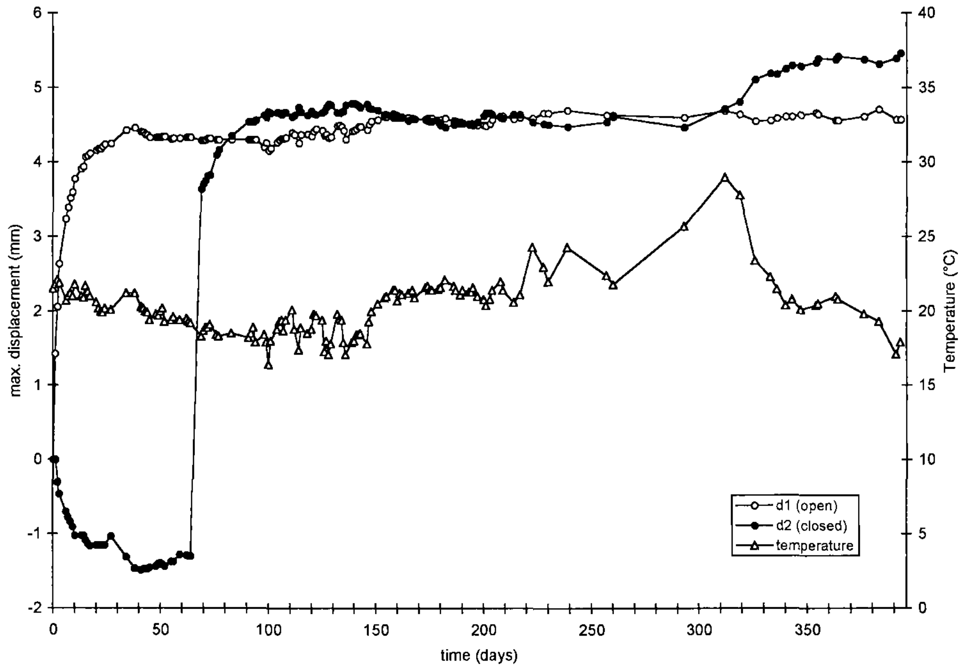

Figure 6. Displacement graph for the center of the Plexiglas™ lids in open and closed glass containers.

6) gives the daily measurements of the displacement of the center of the Plexiglas™ lids and the ambient temperature. The readings for the open system show, in the first 3 days, a rapid downward displacement reaching a value 2.64 mm. During the next 30 days the curve gradually levels off around 4.4 mm.

The graph for the closed system has its starting point at the reading of day 1. This first reading of −1.64 mm (downward displacement) is the result for an open (vented) system. From this point on the jar was closed. During the first 34 days the readings show a gradually upward displacement and a stabilization between day 34 to 64 at a value of approximately −1.3 mm. During this 64-day period, the formation of air bubbles underneath the lid was noticed. From day 1 until around the stabilization point near day 34 the air bubbles gradually grew in size. In the next 30-day period the air bubbles slowly diminished in size until they had vanished, with the exception of some larger air pockets at the edge of the jar at day 64. At day 69 a sudden downward displacement was recorded giving a reading of −3.64 mm. This displacement gradually stabilized again around day 100, at a value of approximately −4.5 mm. Between day 295 and day 366 another downward displacement of −0.93 mm was recorded resulting at day 366 in a reading of −5.42 mm.

When comparing the temperature curve with the displacement curves, a relationship can be seen between the temperature readings and the displacement readings of the different systems. However, the measured changes in temperature do not significantly affect the trend of either displacement curve and are therefore not discussed further.

Table 2. Permeability constants (P) for polyethylmetacrylate (PEMA) in ($cm^3/m^2 \cdot day \cdot atm$) from Yasuda and Stannett (1975).

Permeant	Temperature (°C)	P
H_2O	25	21,013
O_2	25	7.55
N_2	25	1.45

Discussion

Absorption and permeability properties of Plexiglas™.—The outcome of the absorption experiment shows that, with formaldehyde and glycerol solutions, water is readily absorbed by Plexiglas™. Because of the presence of polar groups in its chemical structure Plexiglas™ has a relatively high affinity for water compared to a plastic such as polyethylene, which is a hydrocarbon with no hydrophilic substituents. This means that Plexiglas™ has a higher permeability for water. High water permeabilities are generally encountered with polar polymers or where the segmental mobility is high, as for silicon rubber (Barrie 1968). The weight loss of the sample soaked in 100% glycerol was probably caused by the desorption of the water already present in the sample. Pure glycerol is apparently more hydrophilic than Plexiglas™.

In conclusion, the loss of preservative fluid in Plexiglas™ jars in our collection is mainly caused by water in the preservative solution permeating through the Plexiglas™ to the outside. For this reason, in the pressure experiment and warping experiment, only water was used to determine whether or not water loss by absorption and permeation in a sealed Plexiglas™ jar could be responsible for the pressure drop inside the jar and/or warping of its sides.

Negative pressure.—The pressure experiment shows that the loss of water by diffusion causes an increased negative pressure inside the jar with respect to the outside pressure. In our experimental model, after 1 mo a weight force of approximately 10 kg on an area of 10 by 10 cm was present. After approximately 3 mo this negative pressure had almost doubled. Although inside warping of the top and bottom plates of the jar will have a leveling effect on the pressure increase in time, the relatively high negative pressure and the pressure drop must be explained as the results of a dominant and fast absorption of water in the Plexiglas™ in the beginning and a much slower water loss by diffusion later.

The relatively high weight loss in the first period (days 1–110) compared to the second period (days 111–204) does not completely support this idea, because water absorption in the beginning would not be expected to result in weight loss of the Plexiglas™ container, whereas diffusion of the water to the outside environment later would. Therefore, we expected a higher weight loss in the second period than in the first. Experimental error in our test appears too large to allow for a decisive conclusion. However, it is evident that the increase in negative pressure is hardly compensated for by air influx, which can be explained by looking at the permeability constants for water and air of polyethylmetacrylate (PEMA), an acrylic similar to Plexiglas™ (polymethylmetacrylate, PMMA) (Table 2). The value for water is approximately 15,000 times higher than that for nitrogen and 3,000 times higher than that for oxygen, which means that air permeation through these acrylics progresses at a much slower rate than does water permeation.

Because of the negative pressure inside the jar, the glued connections of the jar will be under considerable stress. Van Dam (2000) describes this phenomenon and suggested that the negative pressure can be avoided by placing a valve in the filling hole of the container, which vents the jar automatically in case of a pressure drop.

Warping of Plexiglas™.—In the warping experiment the Plexiglas™ lid of the open (vented) glass container warped to the inside in a relative short period (1 mo) and stayed nearly stable during the remainder of the experiment. We assume that this type of warping is caused by the swelling of the Plexiglas™ resulting from water absorption. Initially, the swelling starts at the inside surface of the Plexiglas™, progressing slowly through to the outside surface. Because of the difference in expansion of the inner swollen layer and outer unswollen layer, the direction of warping is to the inside. When full absorption is reached, there is no longer a difference in expansion between inside and outside layers, and thus we would expect the Plexiglas™ lid to return to its flat state. However, in our experiment the extent of warping did not change because the swollen Plexiglas™ lid was glued to the rim of the glass container, which prevented it from assuming its original form. If this assumption is correct, it would mean that with open, full Plexiglas™ jars, the warping would be temporary, until the maximum water absorption has been reached, because all sides undergo uniform expansion. On the other hand, it seems more likely that, after full absorption, the top layer of the outside surface would swell less as a result of the loss of water to the dryer outside atmosphere. In this case, warping by absorption in full Plexiglas™ jars will remain consistent.

The warping experiment with the closed container showed, contrary to what was expected, a gradual upward displacement of the lid from day 1 until day 35. This could have been caused by the formation of air bubbles underneath the lid, which were primarily dissolved in the water. Another explanation could be the expansion by swelling of the glued Plexiglas™ lid. Because of the incompressible water underneath, this would leave it no other direction to warp than to the outside, resulting in a larger jar volume. Consequently, the air bubbles underneath the lid would increase in size.

The sudden inward collapse of the lid between day 65 and day 70 resulted in a displacement reading close to the reading of the open container. We assume that fluid loss by diffusion or air loss by an undetected small leak in the closed container caused a decrease of the water/air volume to such an extent that the swollen lid could warp to a position identical to that of the open container. Between day 150 and day 300 the displacement readings of the closed and open containers are almost identical and seem to be stable. At this time, there was no evidence that the process of warping in the closed container could be explained by negative pressure inside the jar. However, between day 295 and day 366, the closed container showed another increase of approximately 1 mm downward displacement of the lid. At this time, the closed container showed that, evidently, a pressure drop resulting from the permeation of water through the Plexiglas™ lid resulted in a further increase of the displacement of the lid. Also, the warping in the closed container was not a gradual process, which would give the deformable Plexiglas™

the opportunity to adapt, but a process of relatively long periods of stability disturbed by sudden collapses. Such collapses easily can lead to the sudden bursting of the jar. In fact, in our opinion, fluid-filled sealed Plexiglas™ jars can be seen as ticking time bombs, which can implode at any moment, unexpectedly.

Solutions.—The use of thicker sheet material in constructing the jars does provide for stronger bonds, although it does not prevent negative pressure from occurring. On the contrary, thick-walled Plexiglas™ jars will initially have a higher water loss, caused by the relatively fast process of absorption, and they will have less ability to warp inside to compensate for the increase in negative pressure than will thin-walled jars. On the other hand, the rate of water loss by diffusion of thin-walled jars will be higher. Therefore, neither option will prevent the inevitable cracking of the jars over time.

Because the increase in negative pressure is the most important cause for the extreme warping and cracking of Plexiglas™ jars, the best method would be to prevent a pressure drop inside the jars. The use of a valve, which vents the jar automatically in case of a pressure drop, seems to be good practical solution. A disadvantage of the use of a valve is that the jar loses a part of its function as an oxygen barrier, which may speed up oxidation processes inside the jar (van Dam 2000).

A pressure drop inside the jar can also be avoided when there is no fluid loss by absorption and/or diffusion. When high concentrations of glycerin are used as a preservative fluid, it is possible to use a concentration that will compete with Plexiglas™ to absorb water. Table 1 shows that zero weight change for a Plexiglas™ sample can be found at a glycerol concentration between 67% and 100%. Theoretically, at such an equilibrium there would be no fluid loss, no pressure drop inside the jar, and consequently no warping. For formalin, the same result could be achieved by adding a hygroscopic salt to the solution. A disadvantage of these hydrophilic fluids is that they may also affect the preservation quality of the specimens.

Another method to prevent water loss would be to coat the Plexiglas™ with a substance that makes the jar impermeable to water. The possible use of coatings and hydrophilic preservative fluids should be further investigated.

The best solution is not to use Plexiglas™ jars at all. Despite their fragility, cast rectangular glass jars still seem to be the best alternative because of their impermeability for most chemical substances, including water and oxygen (van Dam 2000). However, at the present time the demand for these jars is very low and in the near future there is a good possibility that they will no longer be commercially available.

CONCLUSIONS

The causes for warping and cracking of sealed Plexiglas™ jars can be attributed to the water absorption and water permeability of Plexiglas™. A relatively fast initial water loss by absorption results in unequal hydration of the thickness of the Plexiglas™, followed by a slower rate of diffusion of water to the outside environment. This results in uneven swelling of the Plexiglas™ sides of the jar and an increase in negative pressure inside the jar, which causes the jar to warp to the inside and finally to crack at the glued joints. Solutions to these problems may be found in the use of a valve, to avoid pressure drop inside the jar, the use of hydrophilic preservative fluids, or perhaps coatings to prevent water loss.

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