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A NOTE ON THE MANUFACTURE AND INSPECTION OF OPTICAL GLASS

By Dr. C. S. Rao, Technical Development Establishment (Instruments & Electronics), Dehra Dun.

It has been universally acknowledged that optical glass is a strategic material of vital importance, both during peace and war. This is particularly so from the point of view of the defence needs of a country and the importance of being self-dependent in a matter of this kind for a large independent country like India cannot perhaps be over-emphasised. As India does not yet possess any internal source of supply of optical glass, it is thought that it might be of some interest to give here a few notes of a practical nature collected by the author personally during his recent visits to some foreign factories including one of world's leading optical glass manufacturers.

Pots for glass Manufacture

The ingredients constituting the mix for any particular type of glass are usually melted to a homogenous mass in an uncovered pot or crucible of fire-clay. The fire-clay used for making the pot must be carefully chosen for its freedom from iron as well as for its ability to withstand going into solution in the molten glass. One source of good variety fire-clay for manufacturers in the United Kingdom is at Stourbridge in the Midlands.

These pots are either hand made by building in stages or cast in wooden moulds on a mass production basis. For the hand-made pots the wet clay as received from the source is fed into a mixer machine which gradually forces it down a tube in the form of a 4″ diameter ' sausage '. The pot itself is built up in stages from this sausage by hand, the height of the pot built per day being only 6″ or 7″ at a time. The pot maker works on 2 to 3 pots at a time and can, therefore, build three large pots in a week. The usual size of these pots is roughly 3′ in diameter and 3′-6″ in height, each being capable of taking about 2000 lbs. of melt. The interior surface of the pots is usually lined with porcelain to avoid any undesirable ingredients from the clay contaminating the glass melt.

The pots are built in an air-conditioned room (68°F and 85% R.H.), free from draught. They are first allowed to dry on the floor until they are sufficiently set and are then hung by steel straps from the ceiling, the bottom surface being at a height of 8 to 9 ft. from the floor. In this position all the surfaces are freely exposed to the atmosphere and are, therefore, uniformly dried. The date of manufacture is marked on each pot and the pot is allowed to dry in the air-conditioned room for a period of not less than 6 months before it is put to use. It usually happens that on an average about 25 per cent of the pots made are found to crack during the drying period. The cracked pots are pulverized, mixed with water and the clay reused. –Pots of very small size required for special melts are made by wooden moulds.

Certain firms consider that the process of hand manufacture of pots is laborious and takes more time in that a pot requires 6 to 8 months to be ready for use, whereas by making them in wooden moulds it is ready for use in as many weeks, and the pots are at the same time of a smoother and a more uniform finish. The moulded pots used by the British firms at the time of the author's visit had no inner lining of porcelain, but they were contemplating to try sellimanite lining shortly. The larger sized pots made by this method have a capacity of approximately 2,600 lbs. for flint glass or 1,500 lbs. for crown glass.

The stirers used for mixing the molten glass are also made from fire-clay by means of wooden moulds using a wooden core. The stirers have an external diameter of about 3½" and are from 2½' to 3½' in length. No porcelain surface coating is given in their case. A pot and two stirers used for one melt are only good enough for a single operation. The open end of the stirer is either threaded or left plain depending upon the fixtures used.

Raw Materials

The purity of raw materials used in the manufacture of optical glass and the care with which they are mixed together in the correct proportions for any particular grade are among the most important factors for successful production. The bulk of raw material required is sand of high grade purity and free from iron content. Glass factories in Britain use natural sand which is obtained in a remarkably pure form from sandstone deposits at Lochaline near Oban in the West coast of Scotland. After acid processing at the works this sand contains less than 40 to 50 parts of iron to one million parts of silica. Further elimination of particles of iron in the sand is carried out by means of magnetic separators. The other raw materials used, for example potassium-nitrate, potash, alumina, boric acid, barium salts, litharge, etc., should also be of a high degree of purity. The requisite proportions of the raw materials required for any particular mixture are accurately weighed in wooden containers and are then mixed thoroughly to form a homogenous mixture in a mixer with a rotating drum. The optimum period for which the mixing operation is carried out is 3 minutes. A longer mixing period, it is said, is found to result in a partial separation of the components.

The clay pots are initially fired in a large refractory brick-lined oven which holds 3 pots at a time. Town gas is used for firing, no air other than that which enters through the holes leading the gas pipes and those used for observation at the top is mixed with the gas. The gas flame is never played directly on to the pot, as this results in cracking, but is all the time played on top of the pot. The temperature is raised at the rate of 200°C per day to 800°C, the pot being thus fired for 4 days. It is then shifted to another oven which is oil fired to a temperature of 1350° to 1400°C and the pot is allowed to attain this temperature in about 4 to 5 hours. (Note : Oil firing is reported to be cheaper than gas firing and is, therefore, resorted to for reasons of economy. Producer gas is cheaper than oil firing but is liable to contaminate the glass and is, therefore, not used). When the pot has attained the temperature of the oven, about 25 to 30 per cent by weight of cullet is added to the pot and is first allowed to melt (at about 1450°C for Crown glass and 1350°C for Flint glass). The cullet melts completely in about 4 hours' time. Then the mix is added in the form of a cone shaped heap at the centre of the molten cullet at the bottom of the pot. Molten glass has less tendency to adhere to the pot surface than the mix and when the mix is added in the above manner the molten glass at the bottom rises along the sides of the pot and forms a protective layer. After the first lot of mix has melted to glass in about 5 to 6 hours, further quantities of mix are similarly added at intervals and melted down until the pot is nearly full. Then the temperature of the oven is increased by about 50°C and the stirer is introduced into the pot by screwing on to its holder. This is a little tricky operation and requires to be done with experience and care. The raised temperature (1450°C) is called stirring temperature and at this temperature the molten liquid glass is continuously stirred at about 20 to 24 revolutions of the stirer per minute for about 5 hours. The stirer practically reaches to the bottom of the pot; in fact, at the time of insertion of the stirer, it is lowered until the bottom is touched and then slightly raised to relieve it, with the result that the whole mass of molten glass is effectively stirred. Throughout this process frequent readings of the oven temperature are recorded by means of an optical pyrometer.

At the end of the stirring period the temperature is slowly lowered, the rate of stirring is correspondingly reduced and when the melt is fairly viscous to prevent any more stirring, the pot is taken out of the oven and allowed to cool for about 1½ hours, when the outer surface partially solidifies, although still appearing red hot. It is important to remember that the stirring is never stopped completely as long as the pot is in the oven. When the outer surface of the melt has partially solidified, it is covered with a thick layer of sand and the pot and its contents are allowed to cool for the next 4 or 5 days.

During the entire process of the heating of the pot and the molten glass therein the pot is held in a holder which is in the form of a girdle made of thick iron strip. This girdle holder serves to lift the pot by crane and also prevents breakage of the pot. The girdle is slightly loose on the pot with not too much clearance.

Before the pot is completely broken, a sample piece of glass is taken from the top and examined for μ and v values. This is meant to be a rough check and if the values obtained are near about the standard values for this type of glass, the pot is broken up and the glass collected in pieces, large or small as the case may be. The percentage recovery figures in one modern factory are said to be roughly 85 per cent by weight of the melt or 75 per cent by weight of the initial mixture.

Special glass, usually required in small quantities, is melted in platinum crucibles.

The broken pieces of glass are then rough ground to remove sharp edges and awkward protrusions (so as to avoid folds or feathers during subsequent moulding) as well as any visible faults (like stones etc.) near the edges. The pieces are then moulded into slabs of the requisite size. The hot slabs after moulding are rough annealed in a lehr for six hours at the appropriate annealing temperature for the particular type of glass. It is approximately 560°C for crown and 440°C for flint glass. The slabs are subsequently rough ground and two of the opposite sides are polished. The block is then examined for veins or striae, bubbles, seeds, stones, etc.

Glass moulding methods

The procedure adopted is somewhat different in different firms. In one factory, the clear pieces of glass as collected from the broken pot are sent to the moulding department. Here, according to the sizes of prisms or lenses required, the pieces are sorted out, weighed against a moulded specimen and the assorted pieces sent to the moulder. The moulder uses a furnace fired with gas-air mixture for heating the pieces which are kept on a $\frac{1}{4}$" thick iron plate dusted with kaolin to prevent adhesion of the glass to the plate. The glass is allowed to soften to a plastic condition when it is roughly pressed to the desired shape by means of a pair of long flat-ended tongs, is then further manipulated in shape with a long flat-ended tool by which it is finally transferred to the appropriate mould operated by heavy air pressure. It is pressed to the required shape in the mould, ejected, allowed to cool for about half a minute and then transferred to a closed brick chamber where a day's moulded products are allowed to cool slowly for about 24 hours before they are taken out and sent for examination. This firm moulds prisms to approximate shape, but, in the case of lenses, only circular blanks of uniform thickness are moulded which are later ground and finished to the required curvature.

In another factory of repute, any odd piece of glass direct from the broken pot, although it might be of approximately the correct weight, is never taken for moulding to shape as described earlier. Two reasons obviously guide this procedure ; for one thing, faults in glass are not usually detectable in odd pieces ; for another they are liable to give feathers on moulding owing to irregular shapes. In this factory a large slab of glass of the appropriate thickness is taken and ruled on the surface into small squares or rectangles of a size that would give the correct weight for the moulded piece. By hammering on a wooden wedge placed along the ruled line the slab is broken into small cubes. Each small cube is examined for defects, weighed to ensure the correct weight and used for subsequent moulding.

It is best that, during the moulding operation, it should not be necessary to hand press the hot plastic piece with tongs to get the desired shape for moulding. The original piece itself should, as far as possible be of the right shape and size. This is again one of the reasons why odd-shaped pieces from the pot are not directly used for moulding components.

The moulding ovens are gas-fired. For moulding large slabs, the glass pieces are heated in three separate stages in three consecutive ovens, adjacent to one another, so that in the final oven the glass is red hot and is in a soft condition ready for moulding in the tool which is itself heated by a gas ring burner.

In the ovens, the pieces are heated over sellimanite slabs dusted periodically with a mixture of fine china clay and graphite. Before each periodical dusting, the hot slab is taken out of the oven, scraped, dusted with a fresh layer and placed back in the oven. This prevents sticking of the plastic hot glass to the slab, although a thin layer of china clay is finally left on the moulded component.

After moulding, the components are rough annealed as described earlier. The annealed components are washed and examined for any faults.

Annealing of Optical Glass

During the cooling process, the outer portions of a block of hot glass become rigid before the inner, thereby introducing outer layer compression stresses with corresponding inner tensible forces. Owing to the poor thermal conductivity of glass, those stresses can be quite large and, therefore, of serious optical significance. Good and efficient annealing is, therefore, essential.

In one factory, large pieces of glass are ordinarily annealed in a static chamber. They are covered with sand and an iron hood is placed on top. The chamber is electrically heated and maintained steady at a temperature of 550°C for 8 days. It is then allowed to cool down slowly for the next 20 days by cutting off the current. For smaller pieces an electrically operated vertical annealing lehr is employed. The pieces are packed in sand in single cylindrical cast iron vessels, the vessels being piled one on top of the other. By this means it can be so arranged that a batch of perfectly annealed pieces is made available every day by an almost automatic process.

Another reputable firm visited by the author mentioned that they attached great importance to their final fine annealing process for glass parts, as this contributed materially to the ultimate success in producing a strainless material of uniform μ. The glass slabs or moulded parts are placed in cylindrical iron trays of annular shape with an opening in the centre and are covered with sand. A number of such trays are placed one on top of the other in a single annealing chamber. A fan at the bottom of the chamber is used to circulate air through the central hole and over the sides and maintains a uniform temperature throughout. The chamber is electrically heated to the annealing temperature where it is kept for 24 hours. At the end of this period the temperature is allowed to drop at the rate of 20°C per day for the next 5 days ; and thereafter the rate of cooling is more rapid as no more strain can result. A rigid temperature control is maintained throughout the annealing process.

Inspection of Optical Glass

(A) Pieces of glass prior to moulding are examined for the following :—

- (*i*) Correct weight.
- (*ii*) Correct shape.
- (*iii*) Presence of stones, seeds and bubbles.

(B) After moulding, the slabs or components are examined for :—

- (*i*) Presence of veins or striae in glass.
- (*ii*) Seeds and bubbles.
- (*iii*) Feathers or folds introduced during the moulding operation.
- (*iv*) Surface or edge cracks due to contact with a cold tool.
- (*v*) Felps or cracks extending through the material.
- (*vi*) Strain.

Faults enumerated at (*i*) to (*v*) above are examined after wetting the slab or component with a mixture of cedar wood oil and turpentine or naphtha or a transparent colourless varnish of about the same μ value. Veins or striae can be located by projection method using a pointolite source with a pin hole diaphragm, holding the specimen in the path of the light beam and examining the image formed on a light opal or very fine ground glass screen. They can also be located by holding the transparent slab against a distant light source with a dark back ground and examining the slab with a positive lens*.

Seeds, stones and bubbles are located by visual examination of the slab against a bright source of light. Strain in glass can be detected by crossed polarisers, as the double refraction due to strain causes interference which varies in appearance from bright interference colours to patterns in white and black according as the strain is severe or light. Where more accurate results are required for use in instruments of the highest precision, interferomeric methods* of testing the glass are resorted to.

Moulded Components for Optical Instruments

Where extreme precision is the desideratum, as for instance in instruments designed for research, moulded parts are not recommended as the second moulding operation of a piece cut from a previously moulded slab is liable to introduce a little non-homogeneity and therefore a slight non-uniform μ. For such purposes it is therefore preferable to cut and grind the parts from slab. When components are made from slab glass, it will be necessary to make a pre-selection of parts to be cut from the slab in order to avoid seeds and bubbles. With moulded components, on the other hand, such pre-selection is done prior to moulding, and rejections on this score should, therefore, be few and far between. A large 5" base right-angled glass prism was rejected outright by one firm due to the presence of two bubbles of less than a mm. diameter and a third one still smaller.

Usually, glass which is good for freedom from seeds is unsatisfactory owing to the presence of veins, and *vice versa*. Glass with veins is invariably rejected by the producers. The above are production difficulties and it is said that, with certain types of glass, it is practically impossible to obtain glass free from seeds. For special work, however, where choice selection of glass has to be made, a small quantity of any type of glass, free from seeds and veins, it was said, could be supplied at extra cost.

The following table shows the general characteristics of certain common types of optical glass with respect to their liability to the presence of seeds and veins :—

Glass type	*Freedom from seeds*	*Freedom from veins*
Barium flint	Very bad	good
D. B. C.	bad	good
S. B. C.	bad	good
L. F.	good	bad
D. F.	good	not bad
B. S. C.	good	bad
H. C.	medium	good
M. B. C.	medium	medium

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(*See Twyman : ' Prism and Lens Making ', Ch. XI.)