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TRIP REPORT

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TRIP REPORT

Plant Visited - Kimble Glass Co.

Date - January 20, 1955

Trip Made By - H. J. Evans
E. F. Schilling
J. A. Steele

Objective - To be shown frit seal bulb enclosure techniques including processing prior to sealing, and fritted metal-to-glass stud fixtures recently developed by the Kimble Glass Company research groups.

Persons Contacted - Mr. K. Henry - Assistant Director of Research
Mr. J.P. Gleason - Sales Manager
Dr. F.B. Hodgdon - Research Associate - Frit Development
Dr. J.W. Hackett - Director of Research
Dr. C.L. Babcock - Supervisor Glass Physics Research
Dr. J. Francl - Research Associate - Glass Physics
Mr. J.A. Logue - Supervisor Bulb Design
Mr. L.C. Pierzchala - Product Development Engineer
Mr. C.D. Pawlicki - Engineer - Sealing Equipment Development

The morning was spent with Mr. Henry and Dr. F. B. Hodgdon of Kimble and Messrs. Schilling, Evans and Steele of General Electric. The discussion was one acquainting us with their frit development program. They have had 25 to 30 people working for the last six months on their frit program and they have worked with some 80 odd different type frits. Several months ago, they had a lead borate frit which appeared to have the necessary sealing properties. Initial sealing was good but for some inexplicable reason, the frit seal started to leak after four weeks on life test. In the meantime, another frit called #50 was developed which appears to be satisfactory. No. 50 frit is now undergoing extensive testing including life testing of tubes with a frit panel seal, hot and cold thermal shocks and opening and re-sealing tests. The composition by weight of the #50 frit is as follows:

71% PbO, 17% B_2O_3, 9%ZnO, 3%CuO.

<u>Notes of Mr. Henry's Discourse on #50 Frit</u>

The frit has an expansion of 91 - 92 X 10^{-7}, and although it is not suitable for parent glass of the same expansion co-efficient, it works well with a glass that has expansion in the order of 10 X 10^{-7}. This means that in order to use #50 frit, a new glass will be manufactured having the proper expansion co-efficient. As a result of the glass change, the annealing temperature will be increased from the present 440°C to 475°C. It is interesting to note that with the present 90 expansion glass, a permanent deflection of .005" occurs in the center of the face panel when a tube is evacuated. The new glass, with its higher annealing point, will reduce this deformation by a factor of $\frac{1}{13.5}$.

During the frit development program, Kimble attempted to determine whether or not frit at elevated temperatures would exude poisoning contaminants. Kimble vacuum fired a crucible of frit at 450°C for 30 minutes and no noticeable decomposition products were evident. The frit was then fired for five hours at the same temperature and .0002 grams was collected on a condensing plate. The material collected was composed of B_2O_3 and PbO. The writer does not believe that Kimble performed a valid test since contaminants in quantities too small to measure could still have a deleterious effect on cathode life. It was brought out that frit has poor durability in that it has a tendency to weather and decompose in the presence of moisture, acids and other liquids. It is obvious that if such a frit were used, it would be necessary for the tube producer to protect the sealing (fritted) areas during chemical processing including bulb wash. Kimble is working on a rubber gasket which would cover the fritted area with a water tight seal and such a gasket will also protect the surface from mechanical shock or abrasion. They do not have such a gasket at present. They have been using a stripable tape similar to the plastic tape used by electricians.

The lowest temperature at which panel seals have been made by Kimble is 415°C. This temperature is not optimum since the time relationship would be somewhat lengthy but frit sealing at 425 - 430°C appears satisfactory. In the fritted seals, the customary stress induced is in the order of 110 to 150 psi, compression and the same frit used with Corning Glass bulbs induces a compression stress of 1000 psi which is considered too great for satisfactorily sealing yields. This is attributed to the contraction co-efficient of Corning glass which differs by 10 points from that of Kimble.

Kimble has tubes which have #50 fritted panel seals on life test using RCA guns and processing. These tubes have been on life test for six weeks to two months and show no evidence of gas build-up. However, emission has slumped badly and this was explained to Kimble by Dr. Hedrick of RCA as having nothing to do with the frit seal but was caused by improper cathode activation. It is the writer's opinion that this should be taken with a very large grain of salt and when any frit sealing is undertaken by G.E., cathode behavior should be watched very carefully, even if there is no noticeable build-up in gas pressure. Frit panel sealing will necessitate a considerable deviation from the normal practices of the glass companies and tube manufacturers. The deviations from the standpoint of the glass company will more than likely increase bulb cost if this method is considered for monochrome, since most of the deviations are additive. While no bulb costs were discussed, it appears quite safe to assume that a frit sealed color bulb will be competitive with those which are flange sealed. Instead of heat or electro-sealing the face panel to the funnel, which is now the current monochrome bulb practice for closure, the panel and funnel sealing peripheries have to be re-formed so that closer sealing periphery tolerances are obtained. The seal periphery tolerances of present monochrome bulbs would be considerably over ±.100" and frit sealing necessitates no more than ±.025" as measured on the diagonal cross section. After the reforming operation, the sealing surfaces of both the face panel and the funnel are ground to ±.001" using a lapping wheel which is covered with a mixture of water and 320 mesh silicon carbide. This operation takes approximately 20 minutes using Kimble's existing laboratory practice. The frit is applied to the pre-formed ground surface of the cone using a hot dipping process. The ware is pre-heated to 426°C and then the surface is dipped in molten frit which is at a temperature of between 815 and 870°C. When the funnel is removed from the platinum dipping trough, the fritted area is pressed upon a pre-heated metal surface plate which flattens the fritted surface to within ±.007". The funnel is then lehr annealed. A process similar in detail, but different in tooling is used to frit coat the face panel sealing area. It had been determined by Kimble that the flattening of the fritted surface by the metal surface plate enabled them to reduce the sealing temperature by 25°. It is assumed that this temperature reduction was accomplished by having the glass sealing areas in more intimate contact thus permitting earlier vacuum application for the sealing process.

Frit Sealing Demonstration

In the sealing operation, the face panel and cone are aligned mechanically and jigged so that the face panel sealing surface is in contact with, and directly over, the funnel sealing surface. The assembly is placed in an oven and heated to approximately 400°, at which time a Calrod unit, which is concentric to and located approximately ½" away from the panel seal, is turned on and localized heat radiated from the Calrod increases the temperature at the panel seal to approximately 425°C. This heat must be highly localized in order to prevent distortion in the pre-formed parent glass. When the temperature of the panel seal area stabilizes, the bulb is evacuated using a roughing pump, and a vacuum of approximately 26" of mercury is obtained. Due to atmospheric loading on the face panel, the seal area undergoes considerable compression and since the frit is fluid, the two ground surfaces come in very close proximity being separated by approximately .002" of frit. The excess frit is extruded towards the outer edges of the panel seal where it forms a continuous bead. The sealing operation takes place in a matter of minutes (4-5 approximately) and the Calrod units are turned off and the oven temperature cycled down to approximately 400°C. This point would correspond to the temperature plateau that is obtained in normal tube exhaust procedure and processing from this point would be consistent with our present exhaust practice. In the unsealing operation, the process is reversed. It had been determined by Kimble, after lengthy investigation, that it is necessary to apply the vacuum when the bulb is cold and heat the ware up while a vacuum (27" of mercury) is applied. The vacuum is released when the temperature of the panel seal is 410°C as measured on the short axis of the bulb. The baking continues until a general bulb temperature of 426°C is obtained, and a Calrod unit concentric to, and located ½" away from the panel seal, is turned on and the highly localized seal area reaches a temperature of 454°C. As soon as the temperature of the panel seal area stabilizes at 454°C, the funnel is mechanically elevated (in this operation the tube is face down) and a fibrous band of plastic frit appears at the sealing periphery. As the gap between the funnel and panel is increased, these glass fibres, stretched beyond their ultimate limit, separate, and the frit surface tension causes the frit to return to its respective seal area in a somewhat uniform manner. The separated face panel and funnel is cooled down uniformly to room temperature. Several unsealed face panels and funnels were shown on which it was claimed that the sealing and unsealing operation had been performed several times. Kimble claimed that they have no difficulty in sealing at least four times and unsealing three times. Due to the lack of time, we did not see the vacuum test being applied on a bulb with fritted seals, but it is a rather unimportant matter since no high vacuum systems were in evidence.

After witnessing frit enclosure techniques, we were shown a method which had been developed for frit sealing type 430 chrome-iron studs (shaped like large collar buttons) to the inside of the skirt of the face panel. This technique was developed with the idea of using these collar button studs as a means for supporting a shadow mask assembly or an internal sandwich. The stress required to pull these fritted studs from the walls of a face panel using a Tinius Olson universal testing machine was somewhat amazing since on four test samples, the parent glass fractured independently of the frit seal, at loads of 130 - 160 lbs. On one sample the parent glass did not fracture but it required 295 pounds to fracture the stud from the glass and examination of the fracture showed that a sizeable portion of the parent glass had been removed. This verified Kimble's claim that these fritted seals had withstood 250 - 480 lbs. The technique involved pre-heating the face panel to 800°F, transferring it to a precision fixture in which the stud is positioned and heated by small gas fires. An air cylinder action moves the stud forward until the face panel is contacted and then additional force is applied to ensure good stud surface-glass panel contact. The panel is then removed from the fixture and lehr annealed. The author was able to obtain some of the test samples and they are available to interested persons. The frit used for the stud process has a considerably higher softening point so that it would be possible to make a fritted panel seal and still have a stable stud seal.

After the demonstration, we returned to the office of Mr. Henry and were joined by Dr. Hodgdon and Mr. J. A. Logue for a general discussion of what we had seen. During this general discussion, a request was made to Mr. Henry for samples of a glass pan with the face surface to be cylindrical and the two long sides to be concentric with the face. It was agreed that E. F. Schilling would provide the dimensions for such a part and Kimble would undertake to assemble this tray making use of fritting and/or glass sagging techniques. It was brought out that a mold and plunger for pressing a pan of this shape would cost from $3500.00 to $6,000.00 and these costs would be approximately the same for a full face panel similar in shape to the pan. Mr. Henry asked what portion of the mold expense General Electric would underwrite if it became necessary to press the panel. The writer pointed out that we were not qualified to make such a decision but felt that some mutually satisfactory agreement could be reached. It was also pointed out that another group from General Electric would be visiting Kimble in the near future and people in this group would be qualified to discuss costs and financing in greater detail. Since Mr. Henry had indicated that a plunger and mount suitable for pressing the glass pan would be no more expensive than pressing a full face panel, the possibilities of having a cylindrical face panel with the long side being concentric to the face, was discussed. It was brought out that the skirt height of the face panel would have to be shortened considerably so that a relationship of approximately 3/4" existed between the inside of the face and the cylindrically contoured surfaces of the skirt. We were given no assurance that such a configuration could be manufactured practically. The greatest difficulty does not appear to be in the face panel, but with the fluted surface of the funnel, which would have to mate with the face panel surface. We were not given much hope, but Mr. Henry assured us that he would take this up with glass pressing experts. This portion of the conversation was carried primarily by Mr. Evans of the Electronics Laboratory so that no suspicion would be aroused. Mr. Henry felt that there would be some possibility of pressing a funnel to the approximate desired shape and then grinding the surface to the proper dimension. He offered no encouragement whatsoever on the possibility of spinning such a funnel.

Kimble was asked what the probability would be of sealing a family of pre-stressed three mil wires in a fritted panel seal regardless of whether the panel seal contour was flat or cylindrical. Dr. Hodgdon seemed to feel that this could be accomplished with no difficulty and would make an attempt to try sealing wires in such a manner as soon as he received some of the wire that we are presently using. This subject, in addition to cylindrically contoured panel seals or glass pans, should be pursued rigorously when the next delegation from General Electric visits Kimble early in February.

<u>Conclusions:</u>

1. The cost of a frit sealed color bulb would be competitive with any flange type color bulb.

2. Frit sealing of studs or other metallic components capable of supporting an internal sandwich appear feasible.

3. Frit sealing techniques would require deviations from normal monochrome tube processing but there could be some cost advantage to Bulb Preparation processes using a two part bulb.

4. A frit-sealed monochrome bulb will probably be more expensive than the present bulb.

<u>Recommendations:</u>

1. Orders should be placed for frit-bulb components and an investigation similar to one outlined below should be undertaken:

   a. Examine long term vacuum properties of frit sealed bulbs.

   b. Examine, <u>closely</u>, cathode behavior in frit-sealed bulbs.

   c. Investigate adaptability of Kimble's sealing and resealing technique to our <u>monochrome</u> manufacturing processes.

   d. Investigate the use of frit sealed bulbs for our color tube program determining if grille wires can be frit sealed to a ledge or some other anchor.

   e. Examine frit behavior when in contact with moisture, acid, aquadag, aluminum, etc.

   f. Determine the necessity of inert gas flooding for gun protection during the sealing operation.

2. Conduct a cost survey comparing the use of present monochrome bulbs to the advantages and disadvantages of a frit seal type. This analysis should be made when more engineering information concerning the practicality of fritted bulbs is obtained.

3. Cylindrically contoured panel seals concentric to a cylindrical face panel should be discussed in detail during the forthcoming G.E. visit to Kimble.

J A Steele

J. A. Steele
Supv. Tube Mech. Dev. Engineering
CATHODE-RAY TUBE SUB DEPT.

JAS:rl

T-4

TRIP TO OWENS-ILLINOIS LABORATORY, Toledo, Ohio on January 20, 1955

Purpose: To discuss solder glass sealing of color tube bulb funnels and panels.

Those Present:

GE	Owens-Illinois Men Contacted
H. J. Evans	Lewis M. Austin
E. F. Schilling	C. G. Babcock
J. A. Steele	Joe Francl
	John P. Gleason
	James Hackett
	Kenneth Henry
	Frank Hodgson
	Jim Logue
	Leonard Pierzchala

We met with Mr. Henry who is Assistant Director of Research for Owens-Illinois. Mr. Hackett is Director of Research. During various phases of our visit we had occasions to have discussions with the other men listed.

Owens-Illinois has developed a solder glass listed as #50 which they use to seal glass panels and funnels. This glass is 71 per cent PbO, 17 per cent B_2O_3, 9 per cent ZnO and 3 per cent CuO. The glass has an expansion coefficient of $90x10^{-7}$ and a fiber softening point of 405°C. The sealing point is 425°C. At 450°C PbO and B203 begin to vaporize very slowly. There is not any detectable vaporization of the copper or zinc oxides. These would need to be checked to see if bakeout would cause any contamination of a phosphor screen. The solder has poor durability against acid, water or any of the hydroxyl group of chemicals. It is suggested that a rubber thimble be used to protect the solder glass during washing and filming.

During the day we saw panels and funnels dipped in the molten solder glass and the edges pressed flat. Prior to dipping the panels and funnels are heated and reformed so that the edges to be sealed have a standard contour within $\pm$.025". The panel and funnels are sealed by heating the assembly in an oven with additional radiant heat added along the seal line by means of a hot Calrod ring raised to dull red heat and placed about an inch from the seal line. This permits the seal to heat to the sealing temperature of 425°C without heating the panel face to the point where distortion would occur.

Pressure at the seal line is obtained by evacuating the bulb when the sealing temperature is reached. The edges of the base glass are flat so that the separation does not exceed .010". This is assured by grinding the edges before dipping in the solder glass. As the bulb is evacuated the edges come together and the excess solder glass is squeezed out. The Calrod unit is then turned off and the solder glass sets permitting the regular exhaust cycle to be carried out.

If the bulb is to be opened, it first must be heated to near 400°C with the seal line heated to 425°C. Gas is then admitted and a slight positive pressure of one pound per square inch is introduced or the bulb may be heated up side down and gravity used to pull the bulb apart. Seals may be made and reopened three or four times or more in some cases.

Although the coefficient of expansion is 10 points below that of the base bulb glass, stresses after sealing run from 120 to 150 pounds per square inch which is almost neutral. This applies to seals after annealing on Kimble bulbs. Corning glass, which has the same expansion but not the same contraction curve, does not seal well with this solder. They hope to have a solder soon that will seal on Corning glass with 101×10^{-7} expansion.

On Kimble bulbs at room temperature seals using the solder glass have been cycled from 60°F to 160°F at two cycles per hour for a total of 10 to 11 cycles before failure occurs.

Kimble has found that the center of the panel moves in .005 inch when the panel is held for 15 minutes at 440°C plus 30 minutes at 427°C. Kimble is developing a new bulb glass which will have a softening point 30°C higher and should be 13.5 times as stiff at the solder glass sealing temperature.

There is enough distance between the gun and the sealing line of the funnel and bulb to allow the gun to be kept relatively cool until the seal is made. Then the bulb could be evacuated and the gun and neck brought up to temperature to complete the exhaust cycle. This technique coupled with the use of inert gas such as forming gas should eliminate the oxidation of the gun. There still would be an oxidation problem connected with the electron shield if one proves to be necessary.

Kimble has another solder glass which is used to seal on metal lugs. The lugs are 430 stainless steel. The lugs will take over 200 pounds pull perpendicular to the lug at a point 3/8 inch out from the seal. Four of these should allow a 10 pound sandwich assembly to be subjected to a 50 g shock without breakage. Tests in which the seal was held at 450°C for 30 minutes with a lateral pull of 10 pounds on the lug showed a displacement in the lug position of less than .0005 inches.

To the author this trip forced a reappraisal of Kimble and its contribution to the color bulb development program. During the past year the group which originally did research for Kimble has been incorporated into the parent Owens-Illinois Laboratory. This reorganization brought the knowledge and experience of the main glass technologist group to bear on color bulb problems with the result that the above described solder glass was developed. While they admit there are many problems to be solved, results to date indicate that the metal sealing flange can be replaced. The author feels that within the week we should set up a program, preferably in Building 6, in which we evaluate this sealing technique and its consequences on tube quality and cost so that no plans would be initiated to develop factory machinery for processing bulbs with flanges. Owens-Illinois can furnish us with some samples immediately made from monochrome rectangular bulbs.

Since we would receive the panel and funnel already coated with solder glass, we need the following data:

1) An estimate of the bulb cost from Kimble.

2) A cost and feasibility check on a thimble to cover the edge and protect it from corrosion during washing, phosphor deposition, and filming.

3) Life test data to check on gas and cathode emission.

4) Phosphor quality check on bulbs sealed with solder glass.

5) An outlay plan for the alterations in exhaust techniques and equipment with cost analysis for capital outlay and processing.

6) Shipping, handling, and storage shrinkage which might be greater for the glass parts without a metal flange.

7) The problem which may arise from lead vapor if the aquadag or aluminum get on the glass solder and reduce the lead oxide to lead during exhaust.

8) Temperature shock tests.

9) Mechanical shock tests.

10) A comparison with Corning's new flange seal.

We should also strongly encourage Corning to push their glass solder program and see if a glass can be developed which is not subject to erosion by materials containing the hydroxyl ion.

During the discussion Mr. Henry proposed that they make us a bulb with a ledge with the wires sealed in with solder glass. We did not mention that we had such a program now in progress with Corning. It is the author's opinion that Kimble does not know of our program as there was no indication in the proposal that it was a question directed toward securing information. We did state that we had considered such a bulb and if Owens-Illinois wanted to consider it we would like for them to consider a panel and funnel whose sealing boundary would conform to the ledge contours. Mr. Henry agreed to think about it but thought the idea impractical from an economic point of view. It is too difficult to develop the edge on the funnel.

Mr. Henry also expressed a strong desire to establish better liaison between Owens-Illinois and General Electric on future color bulb development programs. We suggested that we proceed to evaluate their solder glass and reexamine our program with respect to removing the metal flange. It was felt that management might come to some decisions on a mutual program during the visit to Toledo planned for February 4th for Dr. Baker, Mr. Lang, Mr. Lee and Dr. Maier.

Howard J. Evans
January 27, 1955

Dist: Those Present and

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E. P. Fischer-Colbrie
G. L. Haller
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