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I-11. Setting Point of a Hard Soldered Ceramics-to-Metal Seal

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I Purpose of Experiment

Several kinds of ceramics are used for the envelope of electronic tubes in which ceramic is sealed to metal by means of hard solder. In case the thermal expansions of ceramic and metal does not match, excess stresses would arise at the seal and reduce its strength and reliability.

Stresses in such seals have not been measured so far owing to the lack of techniques for the measurement. In estimating the amount of stresses it is not certain that whether plastic deformation do take place in the solder below its freezing point or not. Experiments were carried out to measure the setting point of the solder for the purpose of solving the problem.

II Method of Experiment

As direct measurement of stress seemed impossible, indirect method was used.

Bars of alumina ceramic and Kovar metal were sealed together by solder (freezing point: 780°C) under factory condition (Fig. 1).

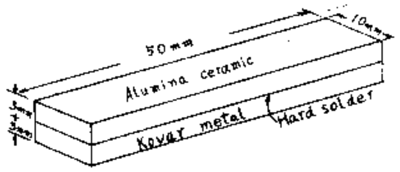

Fig. 1 Ceramic-to-metal seal used as sample for measurement

Thermal expansion of these materials was measured by fused silica differential dilatometer. Result is shown in Fig. 2.

Bending of sealed samples by bi-metallic action during heating or cooling was observed. The method was as follows:

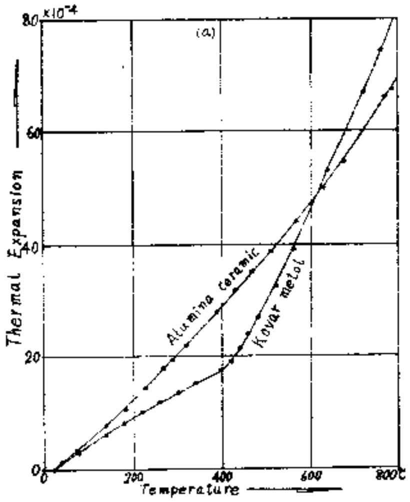

Fig. 2 a)

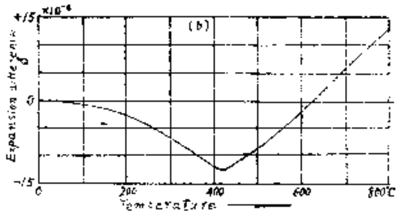

Fig. 2 b)

Fig. 2 a) Thermal expansion curves.
b) Expansion difference.

With fused silica a sample holder was made (Fig. 3-a). In the figure, F is a planely ground surface, A an arm and N a needle point. A sample was clamped between A and F by elastic action of A, facing the surface of ceramic to F. The sample, embraced by the holder, was inserted into a tubular electric furnace (Fig. 3-b) with fused silica window. Hydrogen was slowly introduced into the furnace to prevent oxidation of metal and solder.

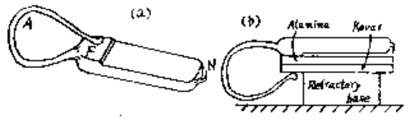

Fig. 3 a) Sample holder.
b) Assembly for measurement.

Temperature of the sample was measured by Pt-Pt13%Rh thermocouple protected by fused silica tube. Deflection of free end of the sample caused by bending was observed by telescope with ocular micrometer (×10), referring to N. One mm of deflection corresponded to 80 divisions of the micrometer.

III Results

III.1 A sample was heated from room temperature to 810°C and then cooled down. Heating and cooling rates above 350°C were 5-7°C/min. Result is shown in Fig. 4. Deflection D was approximately represented by experimental equation $D = K\delta + \text{const}$, where K = 20cm and δ is expansion difference, although some hysteresis phenomena were seen at high temperature. Above 800°C, bending was no more observed.

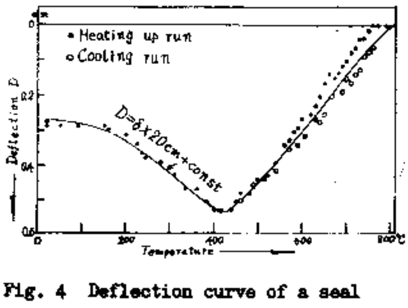

Fig. 4 Deflection curve of a seal during heating and successive cooling

III.2 A sample was heated up to 810°C and then cooled or heated with the rate of 2-3°C/min following schedule of 810°C-720°C-810°C-585°C-810°C. Result is shown in Fig. 5. Hysteresis phenomena

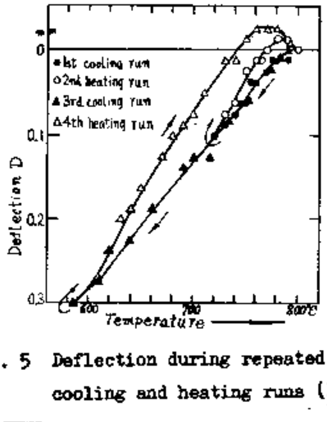

Fig. 5 Deflection during repeated cooling and heating runs (I)

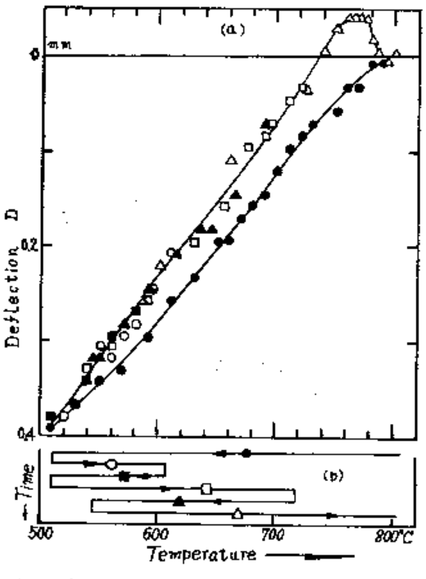

Fig. 6 Deflection during repeated cooling and heating runs (II). b) Time schedule for measurement

were again observed.

III.3 A sample was cooled or heated successively following schedule shown in Fig. 6-b, with the same rate noted above. Deflection of the sample changed as is shown in Fig. 6-a. Except the first cooling run, hysteresis was hardly seen.

IV Discussion

IV.1 Following the theory on bi-metals deflection D of sample is represented by equation: $D = K\delta$, where $K = 25\text{cm}$.1) (Assumed Young's moduli are 2×10^4 kg/mm for Kovar and 4×10^4 kg/mm^2 for alumina.2)) As was shown in III.1, experimental value of K is 20cm. Agreement between these two is rather satisfactory provided that width or length vs thickness ratio of sample was considerably small. The equation $D = K\delta$ fits well with experimental result especially at low temperature. It seems to mean that samples deformed nearly elastically, although slight deviation from elasticity was observed at high temperature.

IV.2 Fig. 5 shows following charges:

1. In temperature vs deflection diagrams, cooling curves (the 1st and the 3rd runs) could be practically be regarded as to go down along an master curve.
2. When sample, after cooling, turned back to heating, hysteresis phenomena were seen. Hysteresis loops were the more distinct the lowere the turning temperature.

And from Fig. 6 it is seen that: 3. Hysteresis phenomena were hardly observed after heating as long as sample was kept in a temperature range not lower and not so higher than the temperature from which the sample was heated.

IV.3 The author attributes the hysteresis phenomena mainly to plasticity of solder, although some other factors might contribute to a little extent. Possible causes of anelastic behaviour of seals are, for example, as follows:

1. As solder has finite thickness, plastic deformation of the solder gives rise anelasticity of the seal.
2. Solder, having fairly high expansion coefficient than both metal and ceramic, solidifies under sevear stress caused by expansion difference with them. This arouse anelastic effects on the solder and the seal.
3. On freezing and melting of solder, there appear many mechanisms such as change of elastic limit with temperature, plastic flow or deformation, crystal growth, work hardening, annealing, appearance and disappearance of lattice defects and so on. These mechanisms interfere with each other and again give rise of anelasticity.

At any rate it seems reasonable to assume that anelastic effects described above act as to diminish elastic stress and deformation of seals. It is also rational to assume that the effects are the less distinct the lower the temperature.

IV.4 Results of experiment can be explained by the assumption noted above as follows:

When a seal is cooled down, solder freezes at melting point M. (Fig. 7).

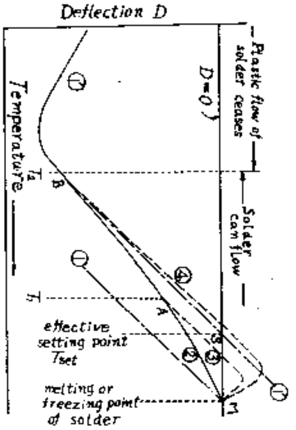

Fig. 7 Schematic representation of temperature-deflection diagrams

If the solidified solder deforms perfectly elastically, deflection D is represented by $D = K\delta + C_1$, where C_1 is a constant, and is shown by curve 1 in the figure. In fact however, the solder deforms plastically releasing part of stress in the seal. Thus $|D|$ is somewhat smaller than $|K\delta + C_1|$ and is shown by curve 2. Curve 2 is a master curve for cooling runs noted in IV.2. At temperatures lower than T , anelastic effects are no more seen experimentally and the seal deforms elastically and reversibly. Here D is represented by $D = K\delta + C_2$ (curve 1'). If extrapolated to higher temperature, curve 1' crosses with line $D = 0$ at S. S is effectively a setting point of the seal (or a equivalent setting point of the seal[3]) and is slightly lower than M.

IV.5 Consider that a sample was cooled to, for example, temperature T_1 and is then heated. Deformation of solder is going to decrease and plastic effects do not come out at least in temperature range not exceedingly higher than T . D is represented by $D = K\delta + C_3$, except at temperatures very near M, where solder flows quickly and D approaches to 0 (curve 3). Similarly, when a sample is heated from T_2, D changes along curve 4. These curves correspond to what are seen in Fig. 4 and 5.

IV.6 As described in the preceding section, D goes along, for example, on curve 4 almost elastically and reversibly at least near point S. This is just what was seen in Fig. 6 experimentally.

IV.7 If the above discussions are right, setting point of the ceramic-to-metal seal can be estimated. Comparing Fig. 4, 5 and 6 with Fig. 7 it is concluded that the setting point is effectively lower than freezing or melting point of solder, but the difference between these two seems not to exceed about 20°C.

IV.8 This difference, however, must depend on many factors, namely; shape and dimension of seal, thickness of solder layer, plastic properties of solder, thermal and elastic properties of ceramics and metal, rates of cooling or heating and so on. Effects of these factors seem to be difficult to calculate theoretically and more detailed studies are needed.

V Conclusion

The effects of the flow of hard solder were investigated in relation to the stress in alumina-to-Kovar seal used in the envelopes of electronic tubes. The ceramics and the metal were sealed together with solder to form a bi-metallic strip to measure the bending during heating and cooling.

Detailed examination on temperature vs bending relation during repeated cooling or heating showed some kind of hysteresis phenomena near the melting point of the solder.

Assuming that these were caused by the flow of the solder, by which some of the stress in the seal is released, the hysteresis phenomena could be explained quantitatively with a result that the equivalent setting point of the seal was found to be about 20°C lower than the melting point of the solder.

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