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SEALING COMPOUNDS

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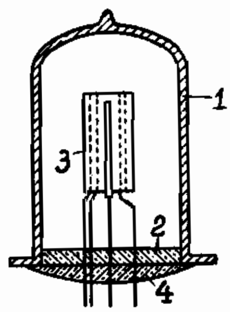

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ALIEN PROPERTY CUSTODIAN

SEALING COMPOUNDS

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This invention relates to sealing material, such as employed for sealing the joints of electric discharge vessels, for instance, in order to secure vacuum-tightness thereof. An example of such a discharge vessel is shown in the accompanying drawing which represents a sectional view of an electron tube. **1** denotes a bulb of high-melting metal, **2** a ceramic cover for this bulb, **3** an electrode system mounted on this cover by means of the leading-in wires, and **4** indicates a layer of glass. Layer **4** is arranged to cover both the body **2** and a flange formed integral with the bulb **1**, and thus seals the joint of the parts **1**, **2**. In the case represented by way of example the layer **4** is utilized also for sealing the leading-in wires into the cover **2**.

Layer **4** is in general made of a hard vitreous material which is difficult to soften and has a low coefficient of expansion, thus being able well to unite with high-melting metals. A further reason why hard glass seals may be highly heated resides in the fact that on account of their low heat expansion strains arising in them are less than in the case of soft glass which highly expands when heated.

Such hard glasses, however, are not suitable for glazing highly expanding material, as iron or alloyed steel, since the curves of their coefficients of expansion are too much different from those of the metal. The aim should be to employ glass of the kind having expansion curves which greatly accord with those of the metal. In discharge vessels with metal bulb and glass seal the temperature of this seal momentarily rises under the influence of pulsating loads. Such rise of temperature, however, will be quite ineffective if a sealing material, such as a glass-like substance or vitreous material, is employed the transformation temperature of which is so low that the heat expansions of all the materials so interconnected are small in the temperature range between the transformation temperature and the temperature of the surrounding air or so-called ambient or room temperature.

As regards the transformation temperature of the sealing material there is a lower limit depending upon the requirements of service. However, the transformation temperature should not be lower than the temperature which the sealing material may acquire during operation. This requirement is in the first place due to the vapor pressure which is negligible only at temperatures below the transformation temperature, being small enough in this case. In the second place such requirement takes the electric conductivity into account, which rises by jumps whenever the transformation temperature is surpassed. In the third place the said requirement is attributable to the viscosity which rapidly decreases at temperatures above the transformation temperature, whereby the seal may be pressed inward by the influence of the outer air.

Where it is desired to obtain a seal which is of a high load capacity and resistive to heat, and if it is not practicable to make use of hard glass, on account of the coefficient of expansion or the high melting temperature thereof, then resource may be had to seals as provided by the invention, these having a comparatively low transformation temperature and a particularly high elongation at rupture or breaking elongation.

Below the transformation temperature the elasticity of vitreous sealing material of the preferred kind obeys Hook's law. The greater the breaking elongation as compared with the thermal expansion the higher may be the thermal demand on such material. Since strains therein can only form until the transformation temperature is reached the maximum of the thermal expansion is $\delta_{th}=\alpha\Delta T$, where α is the coefficient of expansion while ΔT is the difference between transformation temperature and ambient or room temperature. For instance, an ambient temperature of 30°, a transformation temperature of 350° and a coefficient of expansion of $90\cdot10^{-7}$ will effect the thermal maximum expansion $v=2.88\cdot10^{-3}$. The breaking elongation δ_b depends on the tensile strength Z and the modulus of elasticity E of the sealing means and is

$$\delta_b=\frac{Z}{E}$$

According to the invention it is proposed that the breaking elongation be made as high as possible, being made to be at least half as great as the said maximum of the thermal expansion. Thus, in the case of the cited example the maximum breaking elongation should be about $1.5\cdot10^{-3}$. Such a high value of the breaking elongation may be obtained by an appropriate composition of the sealing material.

In this regard sealing material is suitable that has a low transformation temperature, a relatively high tensile strength and a low modulus of elasticity. A low transformation temperature is in well-known manner obtained by means of plumbic oxide and boric acid. The tensile strength is increased by calcium oxide and barium oxide.

Among prior compounds of this or a similar kind there is an enamel cement whose transformation temperature is about 350° and which is composed of 20% silica, 64% plumbic oxide and 16% borax. This cement, however, has a rather high coefficient of expansion and a low breaking elongation and hence is not always suitable for use as sealing material. In addition it contains borax, that is, does not contain boric acid alone but also sodium oxide, and experience shows that alkali added to glass causes this to be electrically conductive at certain high temperatures.

A suitable sealing compound consists of at least 65% plumbic oxide, at most 15% silica, at least 10% boric acid and at least 3% barium oxide or calcium oxide. Good results have been attained by means of a sealing compound composed as follows: 62% plumbic oxide, 15% silica, 11% boric acid and 5% barium oxide. The coefficient of expansion of this special compound is about $90 \cdot 10^{-7}$ and thus is similar to that of the chromium-iron alloys containing about 5% chromium.

Vitreous sealing compounds as provided by the invention have high coefficients of expansion and thereby render it possible that a non-magnetic iron-nickel alloy of about 28% nickel and 72% iron can be hermetically sealed with their aid. This has not been possible so far on account of the high coefficient of expansion of the iron-nickel alloy.

The novel sealing means may be employed for sealing together metal and metal, metal and ceramic material, ceramic material and ceramic material, or ceramic material and glass.

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