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2,934,736

ELECTRICAL RESISTOR

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Fig. 1

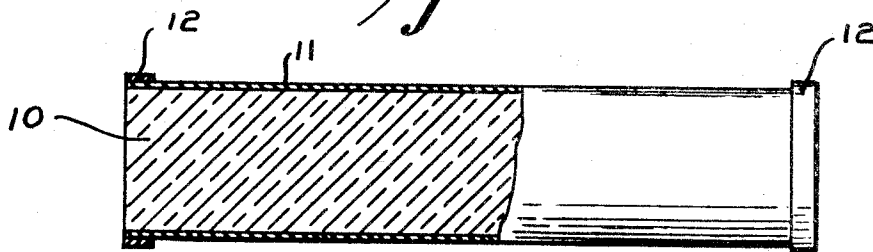


Fig. 2

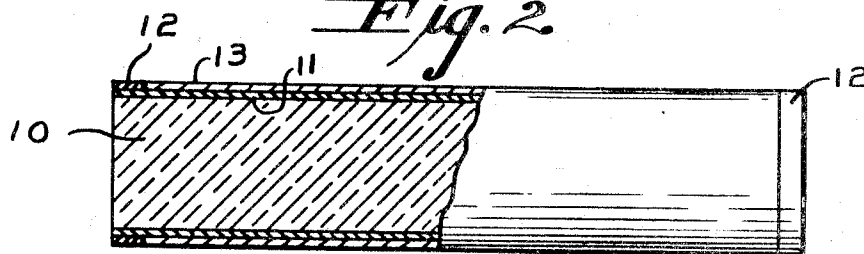
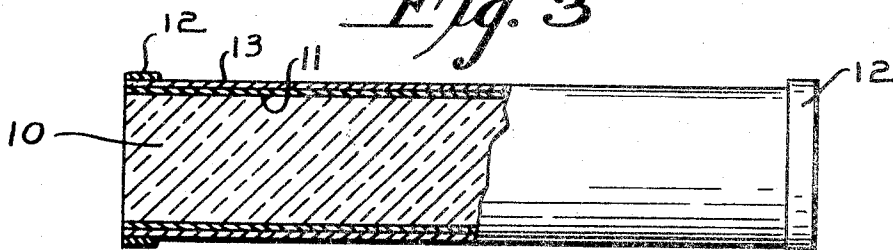


Fig. 3



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2,934,736

ELECTRICAL RESISTOR

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Application October 8, 1957, Serial No. 689,011

6 Claims. (Cl. 338—308)

This invention relates to improvements in electrical resistors of the type comprising a glass body, such as a tube, rod or sheet, an adherent, electroconductive, metal oxide film on the surface of the body, and spaced, electroconductive terminals in electrical contact with the film.

Such resistors are well described in the prior art where it is taught to produce them by heating a glass body, suitably preformed from a glass of any desired type or composition, to a temperature in the neighborhood of 500°–700° C. The heated body is contacted with the vapor or atomized solution of a selected hydrolyzable material to produce on the exposed glass surface a thin, strongly adherent, electroconductive film. Numerous suitable materials and mixtures for producing such films have been disclosed including the chlorides, bromides, iodides, sulfates, nitrates, oxalates, and acetates of tin, indium, cadmium, tin and antimony, tin and indium, or tin and cadmium either with or without a similar hydrolyzable salt or other compound of a modifying metal such as zinc, iron, copper or chromium. The film consists of the corresponding metal oxide or oxides.

The present invention is not concerned with either new film compositions or methods of film production, but rather utilizes any of the known materials and methods. Accordingly, reference is made to the prior art for further details in these respects.

The thickness of the film increases with the length of time the heated body is contacted with the vapor or atomized solution, and electrical resistance of the film generally decreases as its thickness increases. Films having thicknesses from less than the first order of interference colors up to about the tenth order and corresponding electrical resistances of 1,000,000 or more down to 5 or less ohms per unit square can thus be produced. Higher resistances can also be obtained by cutting through a film of a given resistance on a cylindrical body to shape the film into a spiral strip of predetermined width and length.

Resistors comprising electroconductive films of this type provide distinct advantages over other types of resistors for many purposes. However, their electrical resistance tends to be quite unstable, particularly when they are operated under direct current load. This condition manifests itself by a temporary or permanent change in resistance during operation of the resistor. While the tendency is apparent at low temperatures, it becomes progressively aggravated as the temperature involved increases. This circumstance has sharply curtailed use of the film type resistor and has resulted in its exclusion from large areas of the resistor market, particularly applications involving higher operating temperatures.

The electrical instability of film type resistors was initially thought to be associated with chemical changes occasioned by exposure of the film to atmospheric elements such as oxygen or moisture. On the basis of this theory, various types of protective coatings were applied over the conducting film. While most of the conventional coating materials provided little or no benefit in

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this respect, fused ceramic and metal oxide film protective coatings were found to produce marked improvements in electrical stability of the film. However, it soon became apparent that these protective coatings were only a partial solution to the problem and that other factors were also contributing to the electrical instability.

I have now discovered that the presence of an appreciable amount of alkali metal ions in a glass component of a metal oxide film type resistor has a detrimental influence on the stability of the film during resistor operation and that such influence becomes progressively more serious as the operating temperature is increased. The precise manner in which this influence is exerted is not entirely clear as yet. It is known that, under the influence of temperature and direct current electrical load, alkali metal ions migrate within a glass body and that such migration is the result of an electrolytic conduction in contrast to the electronic type of conduction which characterizes the metal oxide films. This would indicate that alkali metal ions, migrating into contact with a film cause a partial alteration of the film and its electrical characteristics due to a chemical or electrochemical reaction occurring between the migrating ions and the film components. In any event, evidence from numerous comparative tests shows that any appreciable content of alkali metal ions in a glass base or other resistor component is detrimental and that, in a relative sense at least, a greatly improved resistor can be produced, regardless of the metal oxide film and/or base glass involved, when a glass substantially free from such ions is employed.

The improved electrical resistor of this invention then comprises a glass body which is substantially free from alkali metal ions, an adherent, electroconductive, metal oxide film on the surface of the body and spaced, electrically conducting terminal members in electrical contact with the film.

Experiments have shown that absolute freedom from alkali metal ions is not a requisite for present purposes. It is also recognized that virtually all glass making raw materials contain varying amounts of alkali metal compounds as impurities and that removal of such impurities is generally impractical if not technically impossible. Accordingly, the term "substantially free," as presently used in the specification and claims, contemplates the presence of such impurities within limits governed by considerations set out below.

The tolerable alkali metal ion content in a glass cannot be precisely stated since it will vary depending on ion mobility in the glass. This in turn depends on the glass viscosity characteristics, the nature of other ions in the glass, and the particular alkali metal ion or ions involved. Thus ion mobility increases with decrease in viscosity. Also lithium ions are much more mobile than sodium or potassium ions. While it is virtually impossible to precisely specify a tolerable level of alkali, it has been found that the content of Na₂O and/or K₂O may in general range up to a half of one percent or so whereas the Li₂O content should be maintained below about 0.05%.

It is well known in the electrical glass art that the electrical resistivity of a glass is essentially a function of alkali metal ion content and mobility. Accordingly, it has been found more convenient, and more meaningful, to specify and control glass quality for present purposes indirectly in terms of glass resistivity rather than directly in terms of maximum tolerable alkali metal ion content. Employing this criteria, the alkali metal ion content in any glass used as a base for resistors in accordance with this invention should be so limited that the resistivity of the glass, determined at 350° C., does not fall below about log 10 that is a value the logarithm of which is ten. One familiar with making electrical glasses and with the measurement of their properties will readily deter-

mine the maximum tolerable alkali content for glasses having different viscosity characteristics.

It has been stated in prior publications that a hazy appearance, said to occur when transparent metal oxide films are deposited on soda-lime glasses containing 10% or more alkali metal oxide, can be minimized by reducing the alkali content of a glass surface prior to filming. This reduction of surface alkali is only a temporary condition, however, since, as is well known, alkali ions migrate in glass when the glass is heated. In fact, coupled with the prior suggestions for alkali removal to reduce haze, are warnings that excess preheating, prior to filming of the glass, may result in a recurrence of the hazy condition due to migration of too much alkali into the surface.

Surface dealkalization is generally inadequate and unsatisfactory as a solution to resistor production problems. Mere reduction in surface alkali to an extent sufficient to eliminate haze will not provide an electrically stable resistor. This is evident from the fact that low alkali glasses, that is glasses having alkali contents on the order of 5% or less, are completely satisfactory insofar as their ability to take a haze-free film is concerned, yet are totally unsuited for stable resistor purposes. Even assuming that a surface could be completely dealkalized, the effect would necessarily be temporary and alkali ion migration during subsequent filming operations, or even during the service life of the resistor, could completely nullify any benefits obtained, although such ion migration might not be sufficient to create any appreciable haze.

The invention is more fully described in conjunction with, and illustrated by, the accompanying drawing in which:

Fig. 1 is a side view partly in section of a cylindrical electrical resistor made in accordance with the invention.

Figs. 2 and 3 are side views partly in section corresponding to Fig. 1 and illustrating further embodiments of the invention.

The resistor of Fig. 1 includes a glass body 10, an electroconductive, metal oxide film 11 deposited on the surface of the body and an electroconductive terminal 12 applied over the film at each end of the body. Film 11 is deposited on the surface of body 10 by a conventional iridizing process in which the glass body is preheated to a temperature of at least 450° C., but not above its softening or deformation point, and preferably to about 600–650° C. The heated body is then contacted by vapors from, or an atomized solution of, the desired metal salt or salts to produce electroconductive film 11. These materials may be anhydrous and fumed onto the body or may be dissolved in compatible organic solvents and applied in solution form. It is usually more convenient, however, to employ an aqueous solution of a salt or salts with sufficient acid in the solution to prevent separation of hydrolysis products. This aqueous solution may then be sprayed on the surface of the heated body to produce the desired film. As indicated earlier a large variety of materials and mixtures have been disclosed as useful for the purpose and any particular film forming material desired may be selected.

Alternatively, the film forming material may be thermally converted into a hot vapor phase to which the body is exposed. In any event, exposure of the body to the film forming material is continued until a film having the desired thickness, and consequently the desired resistance, is formed from the contacting material.

Terminals 12 are desirably of metallic nature and may be formed by well known metallizing procedures. For example, a thin coating of an organo-metal material such as the noble metal resins may be fired on the filmed body. Alternatively, metallizing pastes containing a vitreous flux, such as commercially available silver pastes, may be used. While the terminals are shown superimposed on the electroconductive film 11, they may also be applied directly to the body 10 prior to deposition of

the film if desired. The only requirement is that a suitable electrical connection be established between the film 11 and the terminal 12.

In order to obtain optimum electrical stability an electroconductive film must be deposited on a smooth, non-porous surface; and a vitreous surface is peculiarly adapted. Glass bodies are employed, not only because they provide the required surface and are relatively easy to fabricate, but also because a glass may be varied in composition to obtain the optimum thermal coefficient of expansion. While films can be formed on bodies having widely varying expansion characteristics, as well as rough surfaces, best results for resistor purposes have been achieved with smooth glass bodies in which the glass has a linear coefficient of expansion in the neighborhood of $30-60 \times 10^{-7}/\text{degree C.}$ The glass employed must, of course, withstand the temperature at which the iridizing or filming process is carried out without appreciable softening or deformation.

As previously indicated, atmospheric influences also contribute to the electrical instability of the film. Accordingly, then the present invention contemplates a resistor having an electroconductive metal oxide film formed on an alkali-free glass base and a protective coating superimposed on the film. Such a resistor is illustrated in Fig. 2 wherein a resistor corresponding to that of Fig. 1 has a protective coating 13 superimposed on the electroconductive film 11 and extending between the terminals 12.

In one preferred embodiment of the invention then the improved resistor as described above, has applied over the metal oxide film a fused, vitreous ceramic enamel or glaze covering the film and extending between the terminals, the thermal expansion coefficients of the glass base and the vitreous coating being compatible.

By compatible expansion coefficients I mean expansion coefficients which are similar enough in magnitude so that stresses, particularly surface tensional stresses, sufficient to cause breakage of the resistor or mechanical damage to the electroconductive metal oxide film, when the resistor is heated or cooled, will not be developed either in the ceramic body or the enamel coating.

While it might be expected that the fluxes of the enamel during its fusion would dissolve at least the major portion of the metal oxide film with a corresponding increase in its resistance, I have surprisingly found that such dissolution, if it occurs, is too slight to affect seriously the resistance of the film. Even very thin, high-resistance, first-order films, which are ordinarily unsuitable for resistors on account of their extreme instability, can be glazed in accordance with my invention without disrupting or otherwise destroying them and stable high-resistance films can thus be produced.

In producing a vitreous coated resistor, the resistor film is coated with a layer of finely pulverized ceramic enamel frit, preferably by spraying it with a suspension of the frit in water or other suitable medium. After being dried, the coated resistor is fired at a temperature high enough to fuse the frit without deforming the body. Desirably, firing takes place in a neutral or non-oxidizing atmosphere, such as an atmosphere of nitrogen or argon, since it has been found that, when fired in an atmosphere containing oxygen the resistor tends to become polarized and electrically unstable in use, particularly if the electroconductive metal oxide film is of the first order or thinner. Films of the third order or thicker are not as readily affected and may be fired in air without seriously objectionable results.

The thermal expansion coefficients of the body and the superimposed ceramic enamel should be compatible with each other. The thermal expansion coefficient of the enamel, however, is advantageously somewhat lower than that of the ceramic body because this results in a desirable slight compressional stress in the surface of the

resistor which tends to increase its mechanical and thermal strength.

As indicated earlier, alkali metal ions, if present, can migrate within the protective coating as well as in the supporting body and with a similar detrimental effect on the conducting film. In the interest of electrical stability then it is equally important that any protective coating employed also be substantially free of alkali metal ions.

Alternatively, protective coating 13 may take the form of a metal oxide film applied in the same manner as, and subsequent to, the electroconductive film 12. However, film 13 should preferably have a sufficiently high resistance that the amount of current passing through it between the terminals 12 is relatively small or even insignificant with respect to that passing through film 11.

Fig. 3 illustrates a preferred form of the invention in which a glass body 10 has deposited on its surface an electroconductive metal oxide film 11. A second metal oxide film 13, having a higher resistance, is superimposed on film 11 as a protective coating and terminals 12 are then applied over film 13. By utilizing this type of construction, the two metal oxide films may be deposited successively as part of a continuous coating operation. The electrical resistance of film 13 is desirably sufficiently high to avoid any substantial conductivity between terminals 12, but at the same time must be maintained low enough to permit adequate electrical contact between terminals 12 and film 11 without development of contact resistance or impedance to current flow. It has been found that these conditions may in general be adequately satisfied when film 13 has a resistance of 200 ohms to 10 megohms per square and at least 10 times the resistance of film 11 which may be from 20-10,000 ohms per square. Resistors of this type are described in greater detail and generally claimed in my companion application, Serial No. 537,810 filed September 30, 1955.

As indicated earlier in this specification, a wide variety of materials are known for producing the electroconductive films and any desired material may be employed. However, it has been found that optimum electrical stability is obtained with tin oxide-antimony oxide films. The primary film is preferably a combination of these oxides containing up to 6% antimony oxide whereas a preferred protective metal oxide film, when such is employed, has a higher antimony oxide content on the order of 30-60%.

In the table below a number of glass compositions suitable for use in forming the supporting body are set forth as calculated from the glass batches in weight percent on the oxide basis:

TABLE I

	A	B	C	D	E
SiO ₂	62	62	62	58	44
Al ₂ O ₃	15	15	15	15	7
CaO.....	18	14.7	10	10	-----
MgO.....	5	8.3	7	7	-----
BaO.....	-----	-----	6	6	-----
B ₂ O ₃	-----	-----	-----	4	17
PbO.....	-----	-----	-----	-----	27
ZnO.....	-----	-----	-----	-----	5

Compositions A to D typify RO-Al₂O₃-SiO₂ glasses, that is, glasses consisting essentially of silica, alumina, and alkaline earth metal oxides. Such glasses are particularly suitable for present purposes because their expansion coefficients are in the range of 30-60×10⁻⁷ and their softening points are relatively high, thus permitting film formation at higher temperatures. Glass E, on the other hand, is one customarily used as a protective coating because of its relatively lower softening point. However, it may also be used as a supporting base for carrying out a filming operation at a low temperature, although this is not as desirable because greater

care must be taken to secure a satisfactory film at such lower temperatures.

It should be noted that, while glasses of the type shown above are particularly suited to present purposes, the benefits of the invention may be obtained with any other type of glass, as well, e.g. borosilicate, barium crown, lead silicate, and phosphate glasses. In other words, the term glass is used in a generic sense with reference to the present invention and the benefits of the invention derive primarily from substantial exclusion of alkali metal ions from any base glass rather than from selecting any specific base glass.

The following specific examples further illustrate resistors produced in accordance with the present invention and their marked superiority over prior film type resistors.

Example 1

Glasses A and B of the above table were melted and cane approximately ¼ inch in diameter drawn from such melts. For comparison purposes, cane of like size was drawn from a glass hereafter identified as glass X. Glass X is a commercial glass having the following composition: 60.4% SiO₂, 4.4% B₂O₃, 18.0% Al₂O₃, 7.4% CaO, 8.8% MgO, and 1.0% Na₂O. A piece of each cane approximately 2 inches in length was then heated to a temperature of about 600-650° C. and exposed to an atomized solution of tin and antimony chlorides to produce on its surface an electroconductive film containing about 95% tin oxide and 5% antimony oxide. Film formation was continued in each instance until a film having a resistance of about 60 ohms per square was obtained. Each oxide-coated cane was then provided with metallic terminals as shown in Fig. 1 and a protective glaze, corresponding in composition to glass E of the Table I above, was applied over the film surface.

The resistors thus produced were then set up for testing under a direct current electrical load of 10 watts and with a temperature of about 350-375° C. at the hottest spot on the resistor. At the end of 380 hours continuous operation it was apparent that reasonably stable resistance values had been attained in the resistors produced on the alkali-free glasses and the test was discontinued. It was observed that the resistor employing glass A underwent a maximum change in electrical resistance of 4.9% and the resistor employing glass B a maximum change of 4.7%. These maximum changes occurred well before termination of the test and the resistors thereafter returned to a resistance value closer to the initial value and became relatively stabilized at such intermediate value. Meanwhile, the resistor on glass X had undergone a 12% change at the conclusion of the test and was steadily drifting higher. Thus, the resistance change in each of the resistors employing an alkali-free glass was within a desired limit of 5% specified for a power resistor of this type, whereas the change in the resistor film on a glass containing but 1% Na₂O was well over twice the limit of acceptance and becoming progressively worse with longer operation.

Example 2

Lengths of glass cane dimensionally the same as those described in Example 1 were formed from glass C of the Table I and glass X, the Na₂O-containing glass referred to in Example 1. Resistor films having a resistance of 20-30 ohms per square were laid down on these canes by exposing them to vapors from a solution of tin and antimony chlorides capable of producing a film composition having about 95% tin oxide and 5% antimony oxide. Metal terminals were applied and an organic coating having a silicone base was then applied over the conductive film and between the terminals for protection against abrasion or other physical injury.

These resistors were then placed on test under a direct current electrical load of 8 watts with an operating tem-

perature of approximately 315°. At the end of 312 hours, the test was discontinued and it was found that the resistor on glass X had undergone a change of 8.9% while the resistor formed on the alkali-free glass had changed by only 0.5%.

Example 3

Precision-type resistors were produced on lengths of cane drawn from glass C and glass X in the manner described in Example 2. The conducting film on these resistors was of the same composition as that on the resistors of Examples 1 and 2 but was sufficiently thin that the electrical resistance was in the vicinity of 600–700 ohms per square. The organic coating referred to in Example 2 was applied to these resistors.

Two resistors were placed on test under a direct current electrical load of 2 watts and held on test at an operating temperature of about 145° C. for 500 hours. At the end of this time, it was found that the conducting film on glass X had undergone a change of 2.5% in electrical resistance while the change in the film on the alkali-free glass C was only 0.22%. In this test also the film on the glass containing but 1% Na₂O underwent a change over twice that permitted on precision resistors of this type, while the change in the film on the alkali-free glass was relatively insignificant and well within the acceptable limit.

In accordance with commercial practice, resistors are here referred to as precision or power resistors depending on their electrical load rating and operating temperature. In general, precision resistors are rated at about 2 watts or less and operate at about 200° C. or less, while power resistors have higher load ratings and operating temperature. Resistor specifications vary somewhat, of course, but in most instances the electrical resistance of an acceptable precision type resistor must not change more than 1% from its original value in the course of a prescribed time of test operation, e.g. 500 hours. Power resistors, on the other hand, are usually permitted to vary no more than 5% under electrical load. As indicated by the above data, the present invention now makes possible entry of the film type resistor into the power or high temperature resistor field as well as other areas from which it was previously excluded due to erratic operating characteristics. Because of the improved stability this invention also results in a substantially better film type resistor even in those areas of application where it was hitherto acceptable.

By way of further illustrating the present invention and more particularly its general applicability to alkali-free glasses regardless of other glass constituents, reference is made to a comparative test involving two widely differing base glass compositions, glass 1 being a typical barium crown glass and glass 2 a typical lead borosilicate glass. The compositions as calculated in parts by weight on the oxide basis, are:

TABLE II

	1	2
SiO ₂	50	50
B ₂ O ₃	15	13
P ₂ O ₅		
Al ₂ O ₃	10	7
BaO.....	25	
CaO.....		
MgO.....		30
PbO.....		
ZnO.....		
As ₂ O ₃	0.5	0.4

Two melts were made of each composition with each of the two being identical except that in one melt no alkali metal oxide was intentionally added, while in the second 1.0 part by weight of Na₂O was added. Each melt was analyzed for content of Na₂O, K₂O and Li₂O and the glass resistivity in ohm-cms. measured in terms of log R at 350° C. The results were as follows, the numerals

in each case corresponding to those in Table II and relating to the base glass so identified:

TABLE III

	1-A	1-B	2-A	2-B
Na ₂ O (added).....	0.0	1.0	0.0	1.0
Na ₂ O (analyzed).....	0.008	0.86	0.010	0.95
K ₂ O (analyzed).....	0.004	0.005	0.006	0.011
Log R _{350° C.}	12.260	9.465	11.025	8.275

No Li₂O could be detected in any analysis thus indicating a content below 0.001%.

Glass melted from each of the four compositions was cut and ground into substantially identical, flat, two inch long test pieces. Filmed resistance elements were then produced using three test pieces from each melt, making a total of twelve elements. In preparing these, the glass samples were heated to 650° C. and sprayed with an aqueous solution of a mixture of 95.5 parts SnCl₄·5H₂O and 4.5 parts SbCl₃. The solvent contained 1 part of HCl to 5 parts H₂O. A fourth order green SnO₂-Sb₂O₃ film was produced on each sample having a resistance of 100–125 ohms per square. The filmed glass bodies were then provided with fired on, silver paste terminals on each end and the resistance of each such resistor measured.

The test resistors were subjected to an accelerated 400-hour life test wherein they were operated with a direct current load of about ten watts adjusted to provide a 350° C. "hot spot" or maximum temperature on each resistor at the start of the test. Resistance measurements were made on each sample periodically. The following data was obtained from this test with values given being in terms of percentage change from the original resistance and an average of the three test samples in each instance, and with

R₀=average initial resistance in ohms per square

R₂₄=percent resistance change after 24 hours

R₄₈=percent resistance change after 48 hours

R₄₀₆=percent resistance change after 406 hours.

TABLE IV

	R ₀ (ohms)	R ₂₄	R ₄₈	R ₄₀₆
1-A.....	111.6	Percent -2.7	Percent -3.0	Percent -3.0
1-B.....	115.1	-2.8	-1.6	+6.9
2-A.....	119.1	-2.3	-2.0	-1.5
2-B.....	124.4	-1.5	+0.4	+11.3

It will be noted that all samples underwent an initial decrease of 2–3%. Such a decrease frequently occurs where the sample has had a high temperature heat treatment as occurs in firing of terminals and is of no consequence in the present instance. The significant feature is that the "A" samples underwent relatively minor change after the initial drop. In contrast, the "B" samples, containing 1% Na₂O in the base glass steadily increased to high positive changes which are outside specifications for power resistors.

The effect of alkali is even more striking in a similar set of test samples produced from identical glass samples and film compositions where the samples after life testing were then raised to a 450° C. "hot spot" temperature by increased power load. In these samples after 24 hours the increases in resistance in that time were respectively: 1-A, 2.2%; 1-B, 5.3%; 2-A, 1.2%; 2-B, 11.4%.

The present application is a continuation-in-part of my two copending applications, No. 434,150 filed June 3, 1954; and No. 537,617 filed September 30, 1955, now abandoned.

What is claimed is:

1. An electrical resistor comprising a glass body substantially free from alkali metal ions, an adherent electroconductive metal oxide film on the surface of such

body and spaced, electrically conducting terminal members in electrical contact with the film.

2. The electrical resistor of claim 1 in which the glass body is composed of an alumina silicate glass containing alkaline earth metal oxides and having a thermal coefficient of expansion of about $30-60 \times 10^{-7}/\text{degree C.}$

3. The electrical resistor of claim 1 in which the metal oxide film is composed of tin oxide and up to 6% antimony oxide.

4. An electrical resistor in accordance with claim 1 in which the electroconductive metal oxide film contains an oxide of tin and an oxide of antimony.

5. An electrical resistor comprising a glass body substantially free from alkali metal ions, an adherent electroconductive metal oxide film on the surface of such body, spaced electrically conducting terminal members in electrical contact with the film, and a protective vitreous coating substantially free from alkali metal ions, superimposed on the electroconductive film and extending between the terminals.

6. The electrical resistor of claim 5 in which the metal oxide film is composed of tin oxide and up to 6% antimony oxide.

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