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THE EFFECTS OF POTASSIUM AND SODIUM CONTENTS IN THE CERAMIC SUBSTRATE ON THE LOAD LIVES OF FILM RESISTORS

Yu-Ken Wu, et al

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by

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ABSTRACT: The authors studied the effects of potassium and sodium contents in the porcelain substrate on the service life of film resistors. Cylindrical substrates made of feldspar are used in metallic film and carbon film resistors. The size of a substrate is determined by the load limit and rated power of the film resistor. Substrates are generally polished and roasted at 1000C to give high insulation resistance and chemical stability. Chemical analysis showed that the insulation resistance of ceramic substrates is determined by the alkalimetal content. Load tests on a model RT 1-2 47-kohm carbon resistor at 40C showed that the d-c resistance variation under a d-c loading condition is about 20 times greater than the variation under an a-c loading condition. Alkali metal oxide in the substrate is also responsible for insulation resistance at high temperatures. Tests on a model RJ 2-3 15-kohm metallic film resistor at 70C showed that the initial resistance variation under a d-c load condition is semipermanent and electrochemical corrosion of the film is gradual and relatively long. The authors point out that the service life of film resistors depends on the load power and the contents of alkali metals in the substrate. Experiments on a model RJ metal film resistor (2 w, 80 kohm) conducted by the authors showed that the higher the Na_2O and K_2O content in the substrate, the higher will be the resistance variation and the shorter will be the service life. The resistance stability and service life may be improved by reducing the load power of the film resistors. The authors conclude that the insulation resistance of a substrate varies with the operating temperature. Film resistors with a feldspar substrate will have a higher resistance variation under a d-c loading condition. The resistance variation is reduced with the decreasing alkali metal oxide content. A satisfactory resistance stability under high-temperature load conditions can be obtained by using ceramic material having a low alkali metal content in substrate production. Orig. art. has: 3 tables and 9 figures. English Translation: 14 pages.

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THE EFFECTS OF POTASSIUM AND SODIUM CONTENTS IN THE CERAMIC SUBSTRATE ON THE LOAD LIVES OF FILM RESISTORS

You-gen Wu and Ion-chung Gao

Several film-resistor materials and the general requirements for the surface of the substrate are described. The insulation resistance of several substrates was measured. The effects of various substrates upon the load lives of three kinds of film resistors were investigated. It has been pointed out from the tests of the metal film resistor that the initial changes of the resistance were semipermanent on DC load and the electrochemical corrosion of the films was gradual and relatively long. This effect had much to do with the test conditions and could be slowed down by derating. The different of Na^+ and K^+ in the substrate showed quite different effects.

This paper presents some possible methods for improving the DC aging performance of film resistors. By comparing the load lives of the newer and older substrates, it is considered to be very effective to add alkali-earth metal oxides as the flux for a ceramic substrate.

1. Introduction

A film resistor is a thin film (100 Å-10,000 Å) [1-3] deposited on an insulating substrate with two lead wires connected at the two ends. This film presents resistance to electric current and it has an engraved scale for adjusting the resistivity and a protecting layer over it. There are many substances which can be used for film resistors. We used only three of them for our experiment:

(1) An inexpensive metallic alloy. The alloy was ground to powder and was deposited in a film on a ceramic substrate in a vacuum. It was treated at approximately 600°C to ensure stability. The thickness was in between 500 Å and 1500 Å.

(2) An evaporable saturated organic compound. This compound deposited a glossy carbon film on the ceramic substrate in a high-temperature vacuum. The film possessed physical graphite properties.

(3) Stannous and antimonial ionic solution. The hydrolysis of these solutions on a red-hot substrate (glass or ceramic) formed a metallic oxide film.

The structure and the electrical properties of the film depends mainly on the substance and the deposition technique (degree of vacuum, evaporation rate, thickness of film, etc.) and the surface condition of substrate and its chemical composition. The former concept beyond our scope. The later concept has not been analyzed. From our experimental results we made an analysis on the effects of potassium and sodium contents in the ceramic substrate on the load lives of film resistors.

2. General Requirements of Substrates

2.1. Surface treatment of substrates

The cylindrical substrate of feldspar ceramics is presently used for manufacturing metallic film and carbon film resistors.

The geometrical size of the substrate depends on the characteristics, unit ratio, load and rated power. Because the resistant film is very thin, the characteristics of the resistor depend greatly on the surface condition of the substrate. Closed structure and uniform surface of the substrate are generally required. For the ceramics of the metallic film resistor used in the experiment, the surface had been polished and annealed in high temperatures (about 1000°C) to mirror brightness. The ceramics of the oxide film resistor had been polished too, only the ceramics used for carbon film resistors had both been polished and etched by dilute hydrofluoric acid solution.

2.2. Structure of substrates

As described above, the requirements of the metallic film resistor, carbon film resistor and metallic oxide film resistor were different, but their chemical compositions were the same. Substrates must have insulation resistance and chemical stability in high temperature. Therefore, ceramics are the most suitable substances. Because metallic film resistors have high requirements on the surface conditions of the ceramics, the mineral material (feldspar, quartz, clay, etc.) must be carefully selected. We used four kinds of substrates of the same size. After carefully washing and cleaning, and then adding resistance caps, we measured their insulation resistances at different temperatures. Table 1 presents the chemical analysis results of the contents of alkali metallic oxides in four kinds of substrates.

In Figure 1, several kinds of substrates presented the same degreee of insulation resistance at room temperature. The insulation resistance decreased as the testing temperature increased. Upon comparison with Table 1, we see that the more alkali metalic oxide the substrate contained, the higher the amplitude, the higher the temperature, and insulation resistance decreased. The microscopic structure of the substrates must be clear before any explanation of the cause of the variation in amplitude can be given.

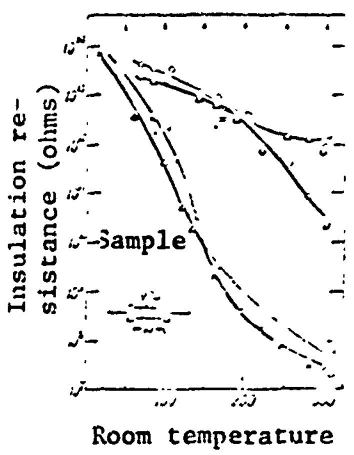

Figure 1. Relationship between insulation and temperature.

Ceramics are composed of a crystal phase, glass phase, and gas phase. Its conductivity mainly depends on the combination layer between the glass crystal, i.e., the transition of alkali metal ions to the glass phase.

TABLE 1.

Substrate / Content, %	Feldspar	Mole	Talc	Glass
K_2O	[illegible]	0.55	[illegible]	[illegible]
Na_2O	[illegible]	[illegible]	0.23	[illegible]
K_2O+Na_2O	[illegible]	[illegible]	0.32	[illegible]

It is generally known that the structure of silicates is a connected lattice structure of Si-O bonds. The radius of an oxygen ion is more than three times greater than that of a silicon ion. The ratio of radii of silicon and oxygen makes a cooperative number of four (4). This determines the tetrahedral $(SiO_4)^{4-}$ to be the fundamental structure unit of silicates.

When an alkali metallic oxide (Na_2O, KO_2O) is introduced into the silicates, oxygen atoms join the crystalline structure. Metallic ions are in the space between the crystalline structure. A monovalent alkali metal ion cannot join together with two oxygen atoms; it then causes the connected lattice structure to break (as shown in Figure 2) [1]. Monovalent alkali metallic ions (K^+, Na^+) have a weak combination force with oxygen. At high temperatures, transition would occur because the excited energy of the ions increases. In a DC field, K^+ and Na^+ move to the negative electrode; this forms ionic conductivity. The transition rate is a function of temperature; of course, when there is more content of alkali metallic oxide (like ordinary glass and feldspar substrate) the volumetric insulation resistance decreases rapidly as the temperature increases.

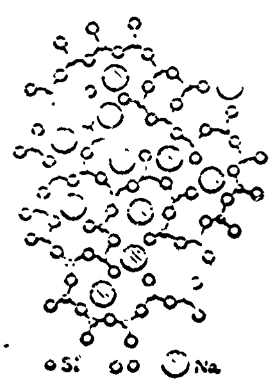

Figure 2. Two-dimensional illustration of how alkali (Na^+) metal ions destroy the continuous lattice structure of silicate.

When a bivalent alkalic-earth metallic oxide (CaO, BaO, MgO) is introduced, the condition is different. Bivalent metallic ions can join together with two oxygen atoms of the tetrahedral and no lattice breakage would occur. Because it has two times as many charges as the alkali metal, the charges are not easily excited and do not easily become loose ions even at high temperature. Therefore, the decreasing of volumetric insulation resistance of talc ceramics and Molestone ceramics still actually depends on the amount of the content of remaining alkali metal (see Table 1).

Through the measurement of the high-temperature insulation resistance of substrates and the analysis of substrate structure, it is not difficult to realize that the volumetric insulation resistance depends on the amount of content of alkali metal. The effects of potassium and sodium ions in the film resistors is an important problem. Although there are some research reports discussing this, the general description is still unclear. For an emphatic discussion of the effect of potassium and sodium ions on the load lives of three kinds of film resistors, we performed the following experiments.

3. The Effect of Potassium and Sodium Contents on the DC Load Lives of Film Resistors

3.1. Load ratio of AC and DC

The type RT, 1W47K Ω film resistor was selected as a sample for the AC-DC load tests and DC load test at one-half rated power, all at a room temperature of 40°C; after 800 hours of testing, we obtained the result as shown in Figure 3. For the same load (1W), the change of DC load resistance is 20 times larger than the AC ones.

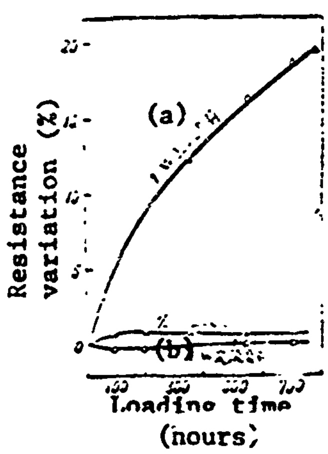

Figure 3. AC and DC loading of carbon film resistors. Key: (a) DC loading; (b) AC loading.

The only factor that introduced this difference was that the types of electric fields were different. According to Joule's law, resistance is a unit of work and the heat of load resistance is independent of the form of electric current (alternating current or direct current). As described in Section 2.2, the contents of alkali metallic oxide have much to do with high-temperature insulating resistance.

Masatugu Shimamune [5] had performed a 50 rated power AC test on a carbon film resistor enclosed tightly in a glass tube. The resistivity remained the same as before after the resistor was cooled down from red hot. For the DC test, the situation was completely different; the resistivity increased irreversibly even for a few times the rated load test for a short period of time. Figure 4 shows the positive and negative terminals of a carbon resistor after being loaded by a direct current. Figure 4a shows the carbon film of the negative terminal side of the engraved line turned from brown to black. After a longer time, the change of color vanished in the middle part and in the positive terminal side; it gathered some of the evaporized alkali metal deposited on the glass wall at the negative terminal as shown in Figure 4b.

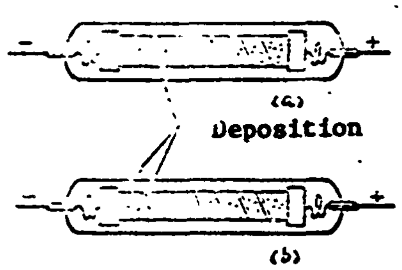

Figure 4. The effects on the positive and negative terminal of a carbon film resistor after DC loading.

We discovered that these phenomena also occurred in metal film resistors and oxide film resistors.

3.2. Test for changing the load polarities

At a room temperature of 70°C, we used a type RJ 2W 15KΩ metal film resistor as sample for a 3W DC load test. Part of the sample's polarities were changed after being loaded for 25, 50, 100, 150, 200 and 300 hours. The result of the experiment is shown in Figure 5.

The experiment indicated: the resistivity variation for normal load increases positively; after the changing of polarities the resistivity varies reversely. The earlier changing of the polarities is closer to initial values of resistivity (as curves 2, 3, etc. in Figure 5). The resistivity presented semipermanent variation during the initial loading period; at this time the thin film was not broken. After

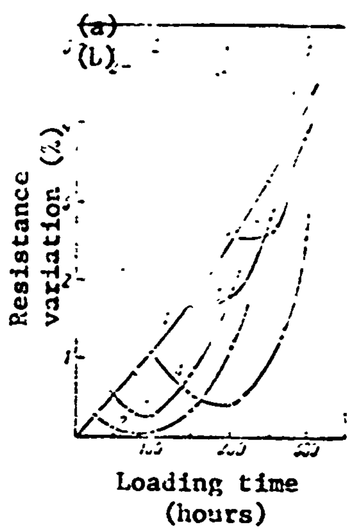

Figure 5. Normal and changing polarities load test. Key: (a) normal; (b) changing polarities.

a comparatively long time (e.g., 250 hours) the resistance did not have the tendency of returning to its initial value by changing more rapidly. Part of the varnish on the negative terminal side of the engraved line dropped off after a long period of loading. The white color deposition on the negative terminal side of the engraved line had proved to be potassium and sodium carbonate.

The curves in Figure 5 show: after changing polarities and continuing the loading, a minimum resistivity variation would appear, and then, as time went on, the variation increased again. Then the longer the time of loading, even if the polarities were changed or not, the film would drop off gradually until an open circuit occurred.

The exact explanation of the above phenomena is to be investigated. From results of experiments we know that there are alkali metal ions liberated from a resistor under direct-current loading.

Paper [5] pointed out that alkali metal ions would form an erosive compound which would cause the carbon film resistivity and negative temperature coefficient to increase. Our experiment used metal film resistors, but ended up with the same result.

At a room temperature of 125°C, we used a type RJ, 24KΩ 2W metal film resistor for 220 V DC for a 200-hour loading test. The result of temperature and average resistivity variation is shown in Table 2.

TABLE 2.

	Temp. coef. (10^{-4}/°C)	Resistance variation (%)
before loading	[illegible]	0
after loading	[illegible]	+4.9

3.3. The effect of alkali metal contents in the substrate on the stability of resistors

In order to find out the effect of alkali metal contents on DC aging of resistors, we selected five feldspar substrates of different contents of Na_2O and K_2O (as in Table 3) to make a type RJ metal film resistor (2W 80 KΩ) for a 300-hour DC loading test. The result indicates (see Figure 6) that the greater the contents of Na_2O and K_2O, the greater the variation of resistivity and the steeper the curves. The reverse is also true.

TABLE 3.

Substrate no. / content, %	1	2	3	4	5
K_2O	2.15	[illegible]	[illegible]	3.76	5.27
Na_2O	0.92	[illegible]	0.97	1.23	1.37
K_2O+Na_2O	3.07	3.59	[illegible]	4.99	6.64

At a room temperature of 125°C, we used a 2W, 16K resistor (No. 3 in Table 3 as substrate) for a 100-hour 3W DC accelerated aging test. From the liberated alkali metal, it was found that Na_2O (average 0.180 mg) was more than four times larger than K_2O ((.029 mg), i.e., the transition ratio of Na^+ is greater than that of K^+: Na^+ has more effect on the breakage of film.

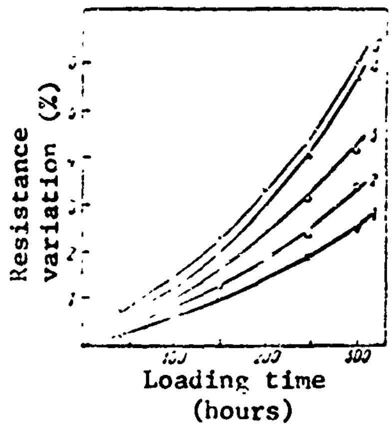

Figure 6. Loading curves of different alkali metal ions contents of substrates of resistors. (1, 2, 3, 4, 5 are curve numbers).

3.4. Load power

Section 2.2 has pointed out that when temperature remains unchanged, if the DC field intensity increases, then the transition rate of ions increases, which means the degree of film destruction increases. Strong field and high temperature resulted in larger irreversible variation and shorter life of resistors. Conversely, suitable decreases of the loading power of resistors would result in higher stability. Therefore, the type RTZ testing carbon film resistor has only half of the loading power of the type RT general-purpose carbon film resistor; the type RJJ precision metal film resistor has only one-fourth of the loading power of the type RJ general-purpose metal film resistor.

From Figure 3 we see that the variation of resistivity is very great in the 1W carbon film resistor DC full-load test; but in the 1/2W load 800-hour test, the resistivity varied very little. Therefore, the stability has a large variation for different loading powers in the same type of resistor. Decreasing the operating power of film resistors is a good way to obtain high stability.

3.5. Research on new substrates

Figures 7, 8 and 9 show the results of the DC load lives test of carbon film resistors made of feldspar, talc and mole ceramics and of metal film resistors and oxide film resistors. From these curves we clearly see that the resistances of the film resistors made of low alkali ceramic substrate varied very little in the whole loading period.

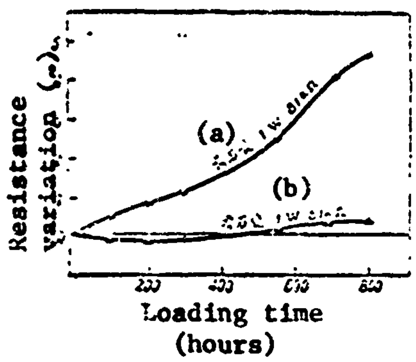

Figure 7. DC loading curves of carbon film resistors of new aged substrates (room temperature = 40°C). Key: (a) talc; (b) feldspar.

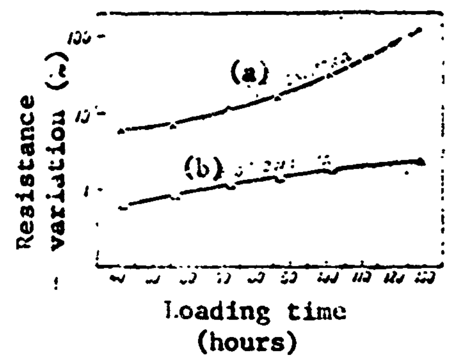

Figure 8. 1000-hour DC loading curves of metal film resistors of new aged substrates (room temperature = 70°C). Key: (a) talc; (b) feldspar.

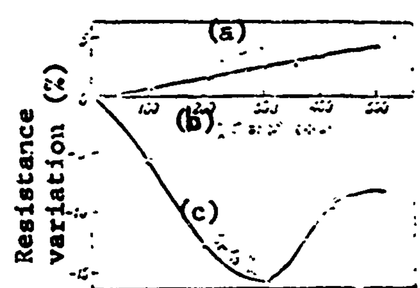

Figure 9. DC loading curves of metal film resistors (2W 32 KΩ) of new aged substrates (room temperature = 70°C). Key: (a) talc; (b) loading time (hours); (c) feldspar.

It is wo thwhile to point out that for the oxide film resistor made of feldspar substrate, the resistance decreased under DC loading, but for metal film and carbon film resistors, it decreased. The change of resistance of the last two film resistors has been discussed in Sections 3.1, 3.2, and 3.3. For the former film, according to paper [6], the resistance decreased under wet loading and the parts of the oxide film of the negative terminal side of the spiral engraved line were reduced.

Tin oxide makes up the main part of the conducting film; it is similiar to a metallic reduction-type semiconductor indicated by SnO_{2-x}. For DC loading, the liberated alkali metallic ions were very active, caused the reduction of tin oxide, and became tin; therefore, in the initial loading time the resistivity decreased rapidly; because the reduced metal film dropped off gradually with time, the resistivity went up again as shown in Figure 9. When talc substrate was used instead of feldspar, the resistivity did not decrease for the whole loading period; this explained that the ionic effect became secondary. The slight oxidization of the film caused the main conducting action, so the resistance increased a little.

From the whole experiment, we know that the improvement of the aging stability of film resistors depends on the selection and the research on new ceramics to take the place of feldspar ceramics.

It is generally very difficult to eliminate the potassium and sodium ions inside the substrates. Feldspar itself contains 12-16% alkali metallic oxides. Using less amounts of feldspar can suitably reduce the content of alkali metals, but it has a certain limit.

Hamura Akira [7] has pointed out that if the content of potassium and sodium oxides of the substrate of carbon film resistors is less than 1.5% of the alkali ions, the transition effects are very insignificant. Although we cannot come to an exact conclusion, we are sure that the less the content of alkali metallic oxide the better. To determine the content of alkali metallic oxides, we not only have to consider the problem of ceramics, but we also must consider the unit ratio of load power and resistance and the actual working conditions. It is better to select potassium feldspar because the transition rate of Na^+ is greater than that of K^+.

Using this ceramic for manufacturing resistors allows the film to be thicker (or have more layers); covering the ceramic with a dielectric film before depositing the resistance film can improve or lower the direct current aging effect.

It is better to use an alkali-earth metal as a flux or as the main part of the ceramics. It can replace the alkali metallic oxide and would not result in technical difficulties in the calcination. The calcination temperature range of talc is very narrow (can be improved by using mineralizers) according to its physical-chemical arrangement. The direct-current aging of resistors made of talc ceramic substrate is very stable. The moisture resistance of talc is not too good, and it has a stronger mechanical strength which makes it difficult to engrave.

Improvement and research on new substrates is the proper way for the direct-current aging of film resistors. It is obvious that this requires the cooperation and extensive research of the substrate manufacturer and resistor manufacturer.

4. Conclusions

1. The volumetric insulating resistance of a substrate decreases as the temperature increases. The amplitude of decreasing depends on the contents of the alkali metallic oxides of the substrates.

2. In film resistors made of feldspar ceramic substrate, the resistivity variation during high-temperature direct-current loading is much greater than that during alternating-current loading. This difference occurs because the liberated alkali metal ions have an erosive and destructive action.

3. For DC loading of film resistors made of feldspar substrate, the resistance decreases as the content of alkali metal oxide decreases. Lowering load power can result in satisfactory stability.

4. The degree of erosion of liberated alkali metallic ions to the film depends on the material of the film: the liberated K^+ and Na^+ in the metal film resistor and carbon film resistor would combine with the film to form erosive compounds causing the resistance to increase; because of the reduction in the oxide film resistor, the resistance of loading decreases in the initial period, and increases gradually afterward. For the metal film resistor, the change of the load polarities can still have the tendency to restore resistance.

5. The less the alkali metal ions the substrate contains, the higher the stability of the DC resistor loading. The effect of Na^+ is greater than K^+. The

amount of K_2O and Na_2O depends on the ratio of loading to unit resistance and the actual operating conditions.

6. The stability of high-temperature loading becomes much better for using new low-alkali ceramics. But talc has one disadvantage, in that suitable ceramics must be selected through compact and complete tests.

Bibliography

1. Burkett, R. H. W.: Recent developments in fixed resistors. *IRE Transactions on Component Parts*, No. 2, p. 112, June, 1959.
2. Burkett, R. H. W.: Tin oxide resistors. *The Journal of the British Institute of Radio Engineering*, Vol. 21, No. 4, p. 301, April, 1961.
3. Burkett, R. H. W.: The performance of pyrolytic carbon. *British Communications and Electronics*, p. 268, 1959.
4. Chan, C. H.: *Inorganic Insulation Materials*, p. 37, July, 1962.
5. Shimamune, M.: The magnetic effects of substrate of carbon film resistors. *IEE*, Vol. 76, No. 1-6, p. 94, 1956.
6. Shirosaka, S. and K. Sugibara: *Component Parts of Electronics*. p. 220, 1960.
7. Hamura, A.: The magnetic effects of substrate and electrical properties of carbon film resistors. *NHK Research*, Vol. 12, No. 1, p. 84, 1960.