JOURNAL OF MATERIALS SCIENCE: MATERIALS IN ELECTRONICS 1 (1990) 61–71

# Review
# Failure mechanisms in bismuth ruthenate resistor systems

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Various failure mechanisms within bismuth ruthernate resistor systems associated with firing, substrate, conductor termination, laser trimming, assembly, packaging, environmental testing, power loading and high voltages, respectively, are reviewed.

## 1. Introduction
The resistor properties which are of relevance to the performance of a thick film hybrid circuit include resistance value, temperature coefficient of resistance (TCR), voltage coefficient of resistance (VCR) and noise coefficient. Deviations in any of these parameters from the designed (desired) values may lead to a failure of the circuit. However, the magnitude of deviation which may cause the failure depends upon the tolerance imposed by the design which, in turn, is decided by the nature of the circuit function (amplification, signalling, filtering, coding, modulating, etc.) and the field of application (consumer, communications, defence, space electronics, etc.). Thus, for instance, in hybrid RC active filters, a deviation in resistance by an order of $\pm 0.5\%$ from the designed value can cause a circuit failure [1]. Similarly, the tolerances allowed in resistor parameters for defence applications are tighter than those allowed for consumer applications.

Deviations in the above resistor parameters from the designed values can occur during any stage of processing (process failures) or during any of the subsequent evironmental, mechanical and electrical tests required for quality conformance approval of the circuit. An understanding of these failures can be quite helpful in recommending corrective actions in the process and thus improving the process yield, quality and reliability of the circuit.

The present paper reviews the failure mechanisms identified by various investigators in a bismuth ruthenate-based resistor system, which is one of the most popular systems used in thick film technology. Failures associated with the following factors have been considered in this review: firing, substrate, termination conductor, laser trimming, component attachment and packaging, mechanical stresses, environmental tests, power loading or refiring, and high voltages.

## 2. Firing
When the screen-printed resistor film is fired through an increasing and decreasing temperature profile, many physical and chemical changes occur in the resistor. These include evaporation of organic solvents, surfactants, burning out of organic vehicle, softening and flowing of glass elements, sintering of functional phase, etc. If the firing parameters, such as peak temperature, soak time, heating rate, cooling rate and furnace atmosphere, are not properly controlled, resistors with required properties cannot be obtained [2, 3].

One specific failure mechanism associated with the bismuth ruthenate resistor system involves the formation of ruthenium dioxide ($RuO_2$) during firing. $RuO_2$ differs considerably from $Bi_2Ru_2O_7$ in its electrical properties as indicated in Table I, and hence the presence of even small amounts of $RuO_2$ can significantly affect the resistance and TCR of fired resistors.

Shah and Hahn [4] carried out X-ray fluorescence spectroscopic and X-ray diffraction analyses on Birox 1400 series resistor materials (Du Pont), before and after firing. They reported the presence of $RuO_2$ in the fired samples of 1411 (10 Ω/□), 1421 (100 Ω/□), 1431 (1 kΩ/□) and 1441 (10 kΩ/□) resistors. The formation of $RuO_2$ during firing was attributed by them to the decomposition of the ruthenate phase at 850° C, where the decomposed products react with the glassy phase. A small decrease in lattice constant of pyrochlore phase on firing, was considered as evidence for the above decomposition process. The following exchange reaction between the decomposed products and glass was envisaged.

$$Bi_2Ru_2O_7 + x(PbO)\text{ glass} \rightarrow \frac{x}{2}(Bi_2O_3)\text{ glass} + Bi_{2-x}Pb_xRu_2O_{7-(x/2)} \quad (1)$$

However, no such decomposition process was reported by either Kusy [5] or Coleman [6]. Kusy [5] studied the X-ray diffraction patterns of resistors printed from a paste consisting of $Bi_2Ru_2O_7$, CdO and lead borosilicate glass, before and after firing. His reports indicate neither a change in lattice constant of $Bi_2Ru_2O_7$ nor the presence of $RuO_2$ in fired samples.

 

TABLE I Electrical properties of $RuO_2$ and $Bi_2Ru_2O_7$ [5]

| Material | Room temperature sheet resistance (Ω/□) | TCR (p.p.m. °C$^{-1}$) |
|---|---|---|
| $RuO_2$ | $4 \times 10^{-5}$ | 5000 |
| $Bi_2Ru_2O_7$ | $2 \times 10^{-4}$ | 0 |

Coleman [6] studied the structural properties of fired Du Pont 1100 series resistors based on $Bi_2Ru_2O_7$. X-ray diffraction studies showed the dried and fired resistors to be identical with same chemical composition, $Bi_2Ru_2O_7$ being one of the constituents. Neither any formation of $RuO_2$ nor any exchange reaction between $Bi_2Ru_2O_7$ and the glassy phase were detected during firing, by Coleman.

Pierce *et al.* [7] investigated in detail various reactions taking place during firing of bismuth ruthenate-based resistors (Du Pont 1400, Birox materials) and reported the formation of $RuO_2$ during firing. During burn-out (200 to 525° C), a portion of ruthenate phase was thought to be reduced to ruthenium metal which subsequently might have been oxidized to $RuO_2$. This was suggested because detailed differential thermal analysis (DTA) and X-ray diffraction studies could not reveal any decomposition of $Bi_2Ru_2O_7$. The initial reduction of ruthenate phase to metal was thought plausible due to reducing conditions prevailing at least at the film/substrate interface, even in a furnace well supplied with air. At the interface, the rate of oxygen transfer would be limited by convection and diffusion which might lead to the absence of an oxidizing atmosphere causing an incomplete combustion of organics in the paste. This might result in reduction to ruthenium metal. Thermogravimetric studies on fine particles of ruthenium and studies on ruthenate-based resistors, where two organic vehicles with different combustion properties were used, supported the above arguments of reduction and reoxidation.

Taketa and Haradome [8] identified the occurrence of strains in thick film resistors as yet another factor which may lead to significant drifts in resistance (of the order of 0.5%) after firing (post-fire drift). Their studies also included resistors based on $Bi_2Ru_2O_7$. The variation of relative resistance of the fired thick film during subsequent annealing at a low temperature, was taken as a measure of the amount of strain occurring along the contact surface between the resistor particle and glass or substrate. It was argued that the above strain would be proportional to the contact surface area and hence the relative resistance variation would be maximum when the metal ($Bi_2Ru_2O_7$)/glass ratio becomes 1 : 1. When the relative variation in resistance was plotted against the glass/metal ratio they obtained a curve shown in Fig. 1 which supported their above arguments. It was also found that increasing the rate of cooling during the firing cycle would introduce a larger amount of strain into the fired film, which might lead to larger drifts in resistance (Fig. 1).

Van Gorp and Van Mourick [9] studied the effect of various organic and inorganic contaminants added deliberately to the furnace atmosphere during firing, on Birox 1300 series resistors. The compressed air

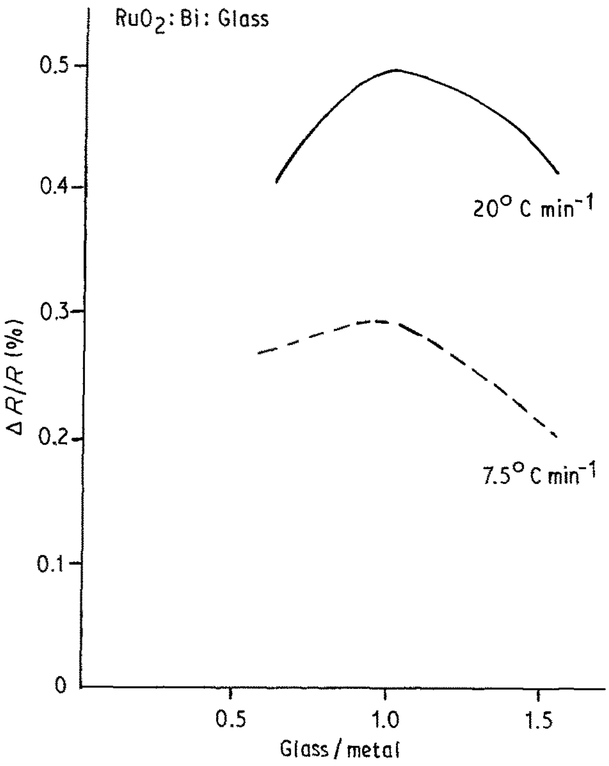


*Figure 1* Relation between $(\Delta R/R)$% and glass/metal, and effect of rate of cooling on these [8].

used for the furnace atmosphere, residual cleaning liquids such as trichloroethylene and some dielectric and glass pastes were identified as potential sources for airborne or particulate contaminants. The effect of various contaminants on the fired resistance of DP 1361, 1341 and 1321 samples is presented in Table II. As shown in Table II, it was found that the high-value resistors were largely affected, resulting in a resistance

TABLE II The influence of 5 mg salt applied on a separate substrate and some co-fired pastes on the resistance of Du Pont 1361, 1341 and 1321 resistors [9]

| Contaminent | DP 1361 | DP 1341 | DP 1321 |
|---|---|---|---|
| KF | + + + | + + | +° |
| $BiF_3$ | + + | | |
| $NH_4Cl$ | + + + | + + | |
| NaCl | + + + | + + + | + |
| KCl | + + + | + + + | + |
| $NiCl_2$ | + + + | + + + | |
| $ZnCl_2$ | + + + | | |
| $BaCl_2$ | + + | + + | |
| KBr | + + + | + + + | + |
| KI | + + + | + + + | +° |
| $MoO_3$ | + + + | + + + | |
| $Sn/InO_x$ | + + | | |
| $H_2SO_4$ | + + + | + + + | 0 |
| $H_3PO_4$ | – | + + + | |
| NaOH | + + + | + + | 0 |
| DP 9061 | −10% | −7% | −4% |
| DP 9911 | −5% | −5% | −10% |
| DP 5217 | −14% | −5% | +3% |
| DP 9101 | +2% | +3% | −8% |
| DP 8190 | −4% | +2% | |
| DP 9950 | +3% | −8% | −12% |
| DP 8315 | +4% | −6% | |
| DP 1361 | −3% | 0 | −2% |

O = positive change, 0 = influence < 5%, + = 5 to 10%, + + = 10 to 25%, + + + = > 25%.

change of more than 25%. Highly volatile contaminants at 850° C such as $H_2SO_4$, $H_3PO_4$, NaOH were found to have a relatively smaller effect on the resistance values. The possible failure mechanism was thought to be the adsorption of conducting material on to the resistor surface which could decrease the resistance value.

An interesting failure case [10] involving erratic resistance and TCR values in early morning and late afternoon production runs was traced to the presence of carbon monoxide and halogen in the furnace atmosphere at these particular hours. Further examination revealed that the intake for the air compressor which was the furnace air supplier, had been located near a parking lot or highway and during rush-hours the air was found to be contaminated by automobile exhaust.

## 3. Substrate

In thick film technology, substrate plays an important role by supporting the circuit, providing a means for mounting various other devices, protecting the circuit from mechanical damage, dissipating heat and providing electrical isolation. However, it can contribute to the failure of a resistor owing to its thermal expansion mismatch and by the degree of its interaction with the resistor material.

Cattaneo *et al.* [11] studied the dependence of electrical properties of Du Pont 1400 Birox resistors on the mismatch between thermal expansion coefficients of the film and substrate. They used 99% beryllia, 96% alumina, steatite alsimag 35 (Type A), steatite alsimag 665 (Type B) and yttria-stabilized zirconia substrates in their investigations. X-ray diffraction and electron microprobe analysis studies on the resistor samples indicated no chemical interaction between resistor and substrate after firing. Although sheet resistances of the films were found to be unaffected by the nature of the substrate, significant changes in the temperature dependence of the resistance were observed. As indicated in Fig. 2, TCR values of the resistors were found to increase with increasing thermal expansion coefficient of the substrate.

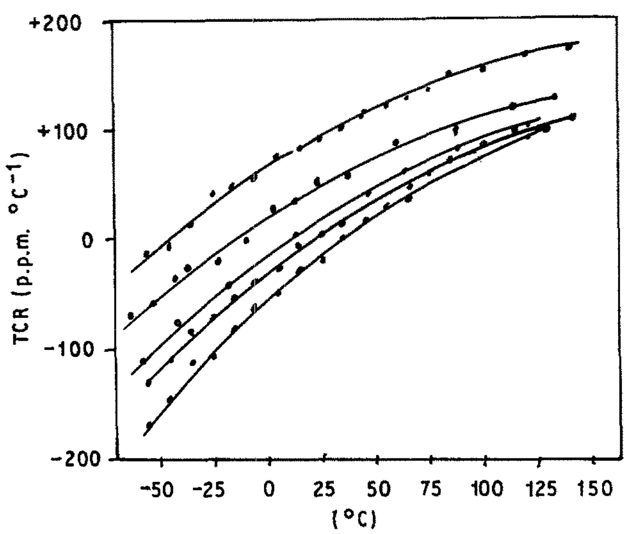


*Figure 2* Experimental TCR values for Du Pont 1400 series resistors screen printed and fired on different ceramic substrates [11].

Further, the temperature, $T_{min}$, at which the TCR changes its sign from positive to negative, was found to shift to lower values with increasing expansion coefficient of the substrate. The above changes in electrical properties of resistors were attributed to the mismatches in thermal expansion coefficients of resistor material and substrate material, respectively.

A substantial increase in TCR values of Du Pont 1461 and 1411 resistors (+160 to +130 p.p.m. $°C^{-1}$) printed on enamelled steel substrates, was reported by Kuzel and Broukal [12]. They compared the above TCR values with those of resistors printed on alumina substrate. The positive increase in TCR value was attributed to the higher expansion coefficient of the enamelled steel substrate than that of the alumina substrate.

A certain amount of chemical interaction between resistor material and substrate would be helpful in decreasing the above mismatches. Shah [13], in his investigations on strain sensitivity of thick film resistors based on $Bi_2Ru_2O_7$ (Du Pont 1400 Birox), found that during firing, $Al_2O_3$ from the substrate dissolved (6 to 8 $\mu$m) into the resistor. This substantial dissolution was found to decrease the thermal mismatch between the fired resistor and substrate and improve the resistor electrical properties. Such an interaction was found to be absent in the case of beryllia substrate, even after repeated firing [14].

## 4. Conducting terminations

The primary function of a conductor material terminating a resistor is to provide an ohmic contact. It is a well-known fact that the contact resistance at the resistor/conductor interface contributes significantly to the overall resistance when the resistors are very short (of the order of $40 \times 10^{-3}$ in ($\sim$0.1 cm) length) [15–20]. A conducting termination may contribute to the resistor failure owing to its migration into the resistor, to chemical reactions between the resistor material and the conductor, or to current crowding when there is a great disparity between resistor and conductor resistivities.

Cattaneo *et al.* [21] studied the influence of metal migration from screen-printed and fired terminations of silver and platinum–gold on the electrical properties of ruthenate resistors. With the help of electron microprobe analysis, the distribution of metal along the length of the resistor was determined. From their studies, they reported that for silver-terminated resistors, the concentration of silver was higher than 0.5% over a distance of 600 $\mu$m from the termination–resistor interface. At the termination the concentration was found to be about 9% to 5%. Along the thickness and width of the resistor the silver content was found to be uniform. From X-ray diffraction studies it was found that at terminations silver was present in large grains (> 2000 nm) while in the resistor it was in smaller grains (50 to 60 nm). This was explained based on the dissolution of large grains of silver in the glassy matrix of the resistor, followed by diffusion and reprecipitation. Silver was found to diffuse without any interaction with the functional phase $Bi_2Ru_2O_7$ of the resistor. In gold–platinum terminated resistors no

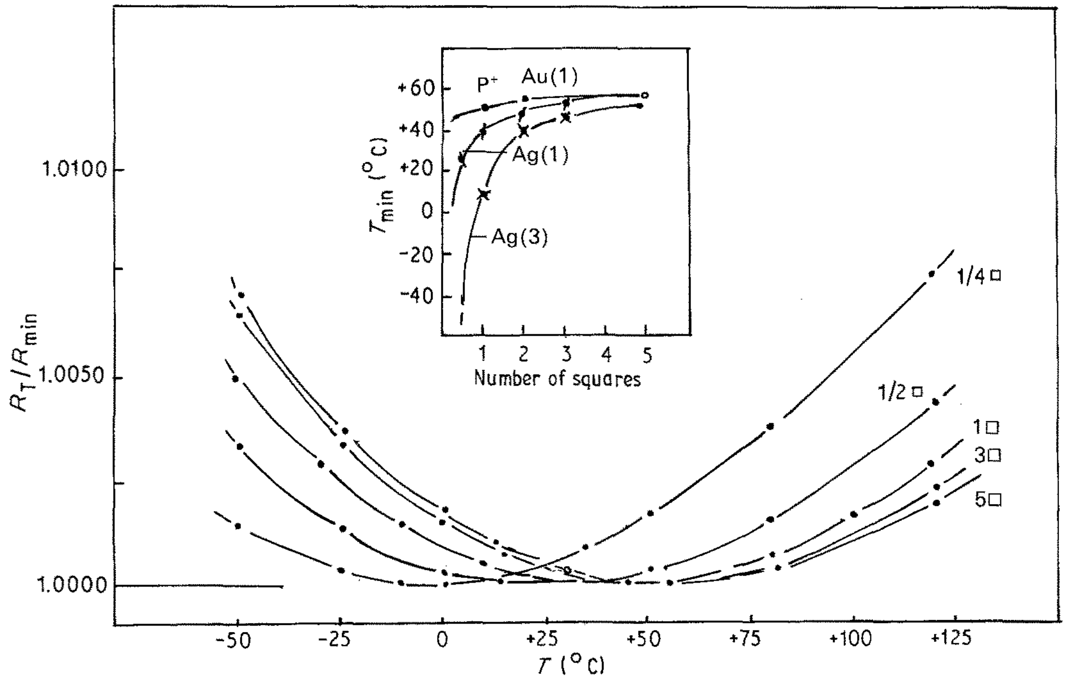


*Figure 3* Normalized resistance against temperature curves for resistors with different aspect ratios and the influence of aspect ratio on $T_{min}$ [21].

diffusion of gold or platinum was detected, except for small resistors with multiple firing. The metal migration was always found to decrease the sheet resistance of the film and shift $T_{min}$ towards lower values. Fig. 3 indicates these variations.

How the incompatibility between resistor and conductor can affect the noise properties of the former is demonstrated in Table III [22]. The high noise figures for 1151 resistors on Pd/Ag conductors were attributed to the poor quality of the resistor/conductor interface.

## 5. Laser trimming

Laser trimming is one of the essential steps in the thick film process and the most convenient method of adjusting the resistance of a fired resistor in large volume production. Despite the general guide-lines for trimming, such as limiting the trimming to within 50% or $8 \times 10^{-3}$ in ($\sim 0.02$ cm), trimming away from termination conductors, maintaining a clean kerf, etc. [23–25], laser trimming can lead to a resistor failure by developing microcracks at the tip of the kerf, by adversely affecting TCR and noise coefficients or by exposing the resistor material and decreasing its environmental stability.

Shah and Berrin [26] carried out post-trim drift studies on Du Pont 1400 Birox resistors. Their studies included the effect of fired resistor thickness, protective overglaze coating, resistor geometry and depth of trim kerf on post-trim drift in resistance. Fig. 4 shows the effect of fired thickness on drift for 100 Ω/□ resistors (1421 Du Pont) with and without overglaze. It was reported that post-trim drift increases with increasing thickness and for an unglazed resistor it became as high as 6.6% for a thickness of $0.10 \times 10^{-3}$ in ($\sim 2 \times 10^{-3}$ cm) over a period of 10 000 h for the 100 Ω/□ resistor. The drift was found to be greater for resistors with thickness greater than $0.5 \times 10^{-3}$ in

TABLE III Noise figures for Du Point 1100 series resistors on various termination materials. Resistor size: 4.5 mm long, 1.5 mm wide [22]

| Conductor material | Conductor mesh size | Noise index (dB) | | |
|---|---|---|---|---|
| | | 1111 | 1131 | 1151 |
| Du Pont Pd/Ag | 325 | −5.6 | −10.3 | +4.0 |
| 8803 | 200 | −11.0 | −5.4 | +6.0 |
| Du Pont Pd/Au | 325 | < −38 | −13.4 | +6.6 |
| 8651 | 200 | < −38 | −13.5 | +5.2 |
| Du Pont Au | 325 | −28.7 | −12.8 | +5.7 |
| 8780 | 200 | −26.7 | −14.2 | +6.9 |
| Du Pont Pt/Ag 8430 | 200 | < −20 | −11.0 | +10 |

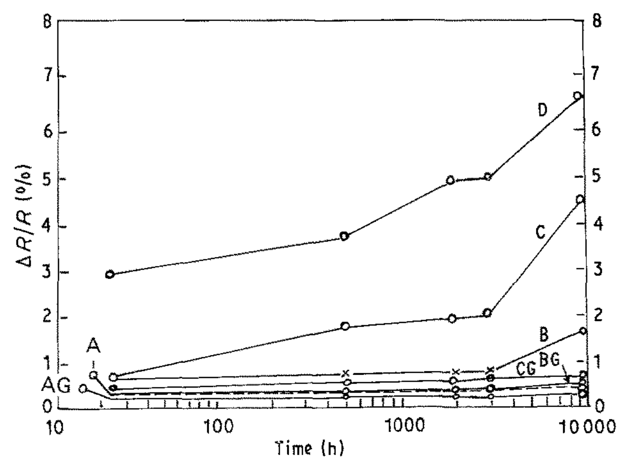


*Figure 4* Effect of resistor fired thickness on drift for 100 Ω/□ resistors, 0.03 in × 0.04 in ($\sim$0.076 cm × $\sim$0.102 cm) resistors Du Pont 1421 ink [26]. A, B, C and D, without overglaze with fixed thickness no. 1, 0.15, 0.17 and 0.22 × $10^{-2}$ cm. AG, BG and CG with overglaze with fixed thickness 0.1, 0.15 and 0.17 × $10^{-2}$ cm.

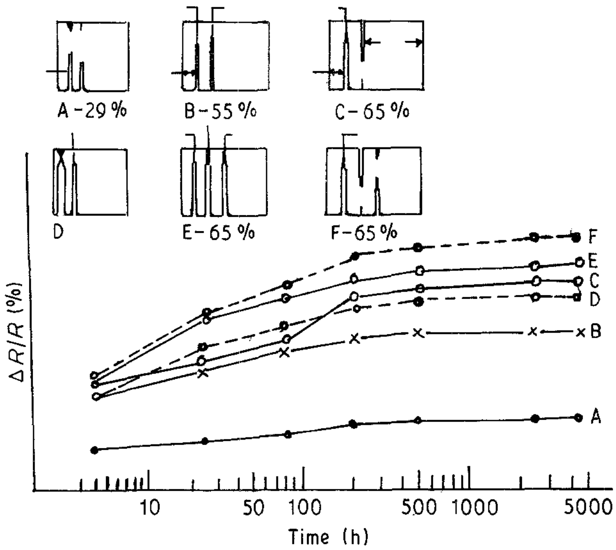


*Figure 5* Effect of trim-cut geometry and per cent trimming on the post-trim drift of 0.07 in × 0.05 in (~0.178 cm × 1.27 cm) in 100 Ω/□ Du Pont 1421 resistors; fired thickness = $0.4 \times 10^{-3}$ in (~$1.02 \times 10^{-3}$ cm) [26].

($1.27 \times 10^{-3}$ cm) and for thickness less than $0.5 \times 10^{-3}$ in ($1.27 \times 10^{-3}$ cm) the drift was within 0.3%. The overglaze was found to decrease the drift significantly. The geometry of the resistor was not found to have much effect on the drift, as long as the thickness was less than $0.5 \times 10^{-3}$ in. The effect of trim-cut geometry and per cent trimming on the drift is shown in Fig. 5. With increasing trim depth, with trim cut close to the conductors, with serpentine cuts, the drift was found to be significant (≃ 3% after 1000 h) even with these resistors (thickness $< 0.5 \times 10^{-3}$ in (~$1.27 \times 10^{-3}$ cm)). From detailed post-trim drift–time plots, Shah and Berrin [26] identified two drift mechanisms, namely short-term drift which is thickness dependent, followed by a thickness-independent long-term drift. The latter was attributed to stress relaxation. SEM studies on the trimmed resistors revealed the formation of microcracks in thick resistors ($> 0.5 \times 10^{-3}$ in (~$1.27 \times 10^{-3}$ cm)) at the kerf tip during trimming. The short-term drift was attributed to the propagation of these microcracks within the film with time. The origin of stresses responsible for long-term drift was attributed to thermal expansion mismatches or to rapid cooling during firing.

Lovati and Bellardo [19] and Rapeli *et al.* [20] studied the effects of resistor geometries (length, width, area) on the post-trim drift behaviour of Du Pont 1400 and 1600 series resistors, respectively. It had been reported [19] that with decreasing resistor area and increasing resistor thickness the post-trim drift in resistance increased significantly irrespective of the sheet resistance of the ink used. When the resistor width and length were decreased to the order of 0.4 mm, the drift at room temperature after 300 h trimming, was more than 1%. By decreasing the thickness of the film from 18 μm to 9 μm the post-trim drift, even in small resistors, could be contained within 0.6%. Similar post-trim behaviour was reported by Rapeli *et al.* from their studies on Du Pont 1600 resistors.

## 6. Component attachment and packaging

A processed thick film substrate is exposed to a wide variety of elevated temperatures during component attachment and packaging. Thus for instance, the substrate will be heated to 400° C for eutectic die bonding involving gold–silicon eutectic bond. It may be heated to about 230° C during solder dipping to tin all the conductors, or for single in line package (SIP), dual in line package (DIP) lead attachment. These temperature effects may cause resistor failures by bringing out significant drifts in resistance due to the earlier process faults, such as microcrack formation in laser trimming, stress formation during cooling, etc. Also, defective packaging may expose the resistors to the environment and may degrade them due to humidity–temperature effects. However, the most important resistor failure mechanism identified in the assembly and packaging stage is chemical degradation of resistors by materials used in this process step.

Coleman [27] studied the effects of various organic resins used in component attachments, packaging and encapsulation on Du Pont 1400 thick film resistors. The resins used were Ablebond 88-2, Epotek H31-D, ESL1110-S and Demetron 6290-00062. After printing and firing of test resistors, the above resins were dispensed on the lids of the packages and were subjected to various curing conditions ranging from 2 h at 120° C to 24 h at 150° C and 3 h at 170° C. This was followed by sealing of the packages in dry nitrogen and the resistors were finally tested for long-term stability (by accelerated life testing). From the studies it was reported that the use of Ablebond 88-2 and Demetron 6290 did not affect the long-term stability much. The other two resins were found to affect the stability significantly. The overglazed resistors were found to change their resistance by an order of magnitude less than the unglazed ones and the change was of the order of 1.4% after 10 000 h. The resistor failures associated with H31-D resin were attributed to the degradation caused by a catalytic action of

TABLE IV Gas analysis of cans containing various resins [27]

| Resin | $H_2O$ | CO | $CO_2$ | $NH_3$ | $CH_4$ | $H_2$ |
|---|---|---|---|---|---|---|
| Empty | 0.63 | 0.01 | 0.03 | 0.01 | – | – |
| ESL (uncured) | High | – | 0.33 | 1.95 | 0.22 | 1.40 |
| ESL (cured) | 1.83 | 0.05 | 0.19 | 0.02 | 0.02 | – |
| H31-D (uncured) | 0.73 | 0.05 | 0.04 | 0.01 | – | – |
| H31-D (cured) | 0.51 | 0.01 | 0.03 | – | – | – |
| Demetron (uncured) | High | 0.07 | 0.79 | 0.71 | 0.20 | 0.19 |
| Demetron (cured) | 1.95 | 0.17 | 0.29 | 0.02 | 0.05 | – |
| Ablebond (uncured) | 12.00 | 0.47 | 1.59 | 5.90 | 0.41 | 0.39 |
| Ablebond (cured) | 2.80 | 0.05 | 0.34 | 0.12 | 0.03 | – |

TABLE V Ageing of DP 1411 for various times at 150° C [27]

| Contaminants | Change in resistance (%) over | | |
|---|---|---|---|
| | 100 h | 1000 h | 9400 h |
| $H_2O$ | 1000 | – | – |
| | 2 | −10 | −14 |
| $CO_2/H_2O$ | 1 | 2 | 3 |
| $NH_3/H_2O$ | 1 | 3 | −2 |
| Formaldehyde (1) | Open circuit within 5 h | | |
| Formaldehyde (2) | 1 | 10 | 17 |
| | 3 | 19 | 33 |
| Butyralactone | 1 | −4 | −16 |
| | −1 | −10 | −20 |
| Phenol | Open circuit within 5 h | | |
| $BF_3$ ethylamine | Open circuit within 5 h | | |
| ESL 1110-S (uncured) | 2 | −3 | −10 |
| | 1 | −5 | −16 |

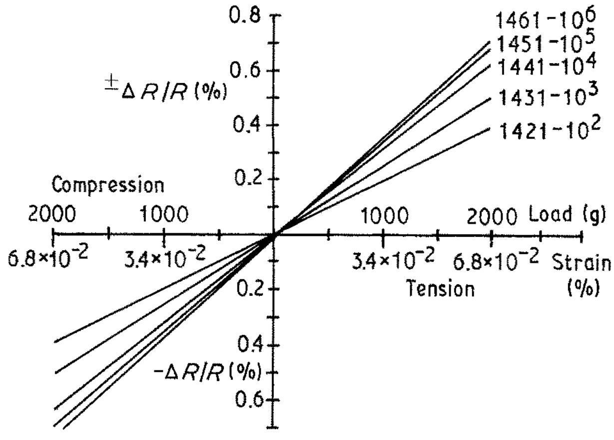


*Figure 6* Summary of strain sensitivity of Du Pont 1400–Birox untrimmed resistors without overglaze [13].

residues resulting from breakdown of the boron trifluoride complex used in the resin. With ESL-1110S, the resistance was found to decrease for unglazed resistors. The factors that may be responsible for the above resistance changes are summarized in Tables IV and V. Table IV shows the type and concentration of gaseous products released from various resins cured and uncured. Table V indicates the influence of these gases individually (added as deliberate contaminants) on the resistance value of resistors. These gases were found to lead to the formation of carbon-rich deposits on the resistor surface, chemical attack on the overglaze material on resistors by potassium, chlorine and lead, etc., and to the ultimate degradation of resistors.

Coleman [27] also investigated the influence of four encapsulant resins Dexter Hyrol ES4281, Morton EPC, ESL 240 SB and Hooker Durez on Du Pont resistors. Unglazed Du Pont 1411 and 1461 resistors were found to be more unstable when coated by epoxide and phenolic resins than when coated by silicone resins. The effect on stability of all the other resistors was found to be similar for all encapsulants. Overglazing with appropriate glasses was found to improve the stability remarkably.

The influence of plastic encapsulation and overglaze on Birox 1300 series resistors was studied by Sinnadurai *et al.* [28]. Their studies consisted of trimmed and untrimmed resistors with and without overglaze and encapsulation. The type of plastic used for encapsulation and the test conditions like thermal and damp-heat overstress with bias to which the resistors were subjected were selected so as to provoke excessive drift and anomalous behaviour. Results of their studies are presented in Table VI. The studies indicate that an unsuitable plastic encapsulant can degrade the ruthenate resistors significantly. The non-encapsulated resistors, in general, were reported to have more stability than the encapsulated ones. Overglazing was found to improve the stability of resistors. The physical interaction between plastic and resistor materials was thought to be responsible for the anomalous behaviour of encapsulated resistors.

## 7. Mechanical stresses

Mechanical stresses may originate during process operations such as mounting, encapsulation, soldering of rigid soldered leads, injection moulding, etc., and also may arise due to thermal expansion mismatches among various materials. Mechanical stresses may also be imposed during reliability tests such as constant acceleration, shock and vibration tests. Resistance changes of the order of 0.5% have been reported due to deflection of the substrate.

Shah [13] carried out detailed investigations on the strain sensitivity of Du Pont 1400, Birox resistors to evaluate the effect of these stresses on the resistance. Strain sensitivity (percentage change of resistance for a given change in applied stress) for all the untrimmed, unglazed resistors was found to be positive under tension and negative under compression. It was found to vary linearly with tension or compression, independent of fired thickness. At a given tension the strain sensitivity was found to increase with sheet resistance of the film. Variations in strain sensitivity with tension or compression were found to be reversible in nature. These aspects are summarized in Fig. 6. The effect of thickness on strain sensitivity was found to be significant once laser trimming was carried out. As shown in Fig. 7, the strain sensitivity of a laser-trimmed unglazed Du Pont 1421 resistor with a thickness of $0.4 \times 10^{-3}$ in ($\sim 1.0 \times 10^{-3}$ cm) was found to vary linearly and reversibly with tension or compression, the change being within $\pm 0.5$. However, once the

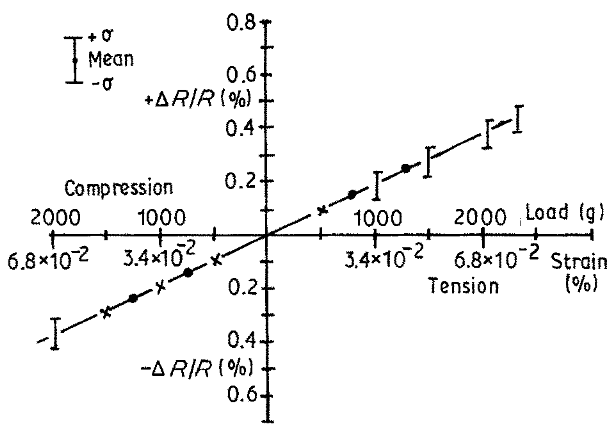


*Figure 7* Strain sensitivity of Du Pont 1421 ($10^2\,\Omega/\square$) unglazed, laser-trimmed material, $t_f = 0.4 \times 10^{-3}$ in ($\sim 1.02 \times 10^{-3}$ cm), five substrates, $N = 30$ resistors [13].

TABLE VI Changes in resistance (to the nearest %) of Du Pont 1300 series resistors at 500 h over stress [28]

| | 170° C | | | | | 150° C | | | | | | | | | 85° C | | | 85° C, 85% r.h. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | No encaps. | Encaps. | | | | No encaps. | Encaps. | | | | | | | | No encaps. | Encaps. | | No encaps. | | Encaps. | | | | | | | |
| | | NG | | G | | | NG | | | | G | | | | | NG | G | NG | G | NG | | | | G | | | |
| | | NT | T | NT | T | | NT | | T | | NT | | T | | | | | | | NT | | T | | NT | | T | |
| $R_A$ (10 Ω/□ paste) | ± | Not biased | | | | ± | + | − | + | − | + | − | + | − | ± | + | − | +− | +− | + | − | + | − | + | − | + | − |
| Nom. trim. 24 Ω to 36 Ω | 0 | −1 | 1 | 0 | 0 | 0 | 2 | −2 | 0 | −3 | 0 | 0 | 0 | 0 | 0 | | | 1 | 0 | 3 | 2 | 5 | 5 | 0 | −2 | 0 | −4 |
| $R_B$ (1 kΩ/□) 3.1 to 4.7 kΩ | 0 | −2 | −2 | 0 | 1 | 0 | −1 | −11 | −1 | −10 | 0 | 0 | 0 | 0 | 0 | 1 | 0 | 1 | 0 | 1 | 3 | 1 | 4 | 0 | −3 | 0 | −8 |
| $R_C$ (10 kΩ/□) 31 kΩ to 47 kΩ | 0 | −6 | −8 | −1 | −1 | 0 | −1 | −16 | −1 | −18 | 0 | 0 | 0 | −0 | 0 | 1 | 0 | 0 | +1 | 1 | 3 | 1 | 3 | 0 | 0 | 0 | −4 |
| $R_D$ 500 kΩ/□ 1.6 MΩ to 2.4 MΩ | 0 | −4 | −2 | 0 | −1 | 0 | 6 | −1 | 0 | −1 | 0 | 0 | 0 | −1 | 0 | | | 0 | −1 | 2 | 4 | 3 | 3 | 0 | 1 | 0 | −7 |
| Average changes of significant groups | 0 | −3 | | 0 | | 0 | +1 | | | −8 | 0 | | | 0 | 0 | 1 | 0 | 1 | 0 | 2 | | | 3 | 0 | | | −3 |

NG = not glazed, G = glazed, NT = no tension, T = tension.

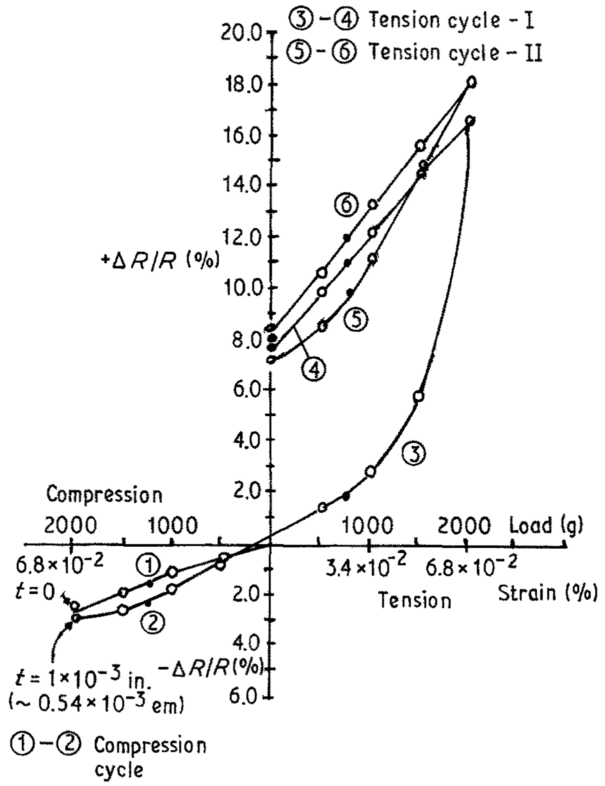


*Figure 8* Strain sensitivity of Du Pont 1421 (100 Ω/□) unglazed, laser trimmed material, $t_f = 0.9 \times 10^{-3}$ in ($\sim 2.28 \times 10^{-3}$ cm), $L = 0.060$ in (0.152 cm), $W = 0.090$ (0.228 cm) [13].

fired thickness was above $0.5 \times 10^{-3}$ ($\sim 1.27 \times 10^{-3}$ cm) and the resistor was subjected to laser trimming, the strain sensitivity was found to vary irreversibly. The magnitude of change was as high as +16 for a tension of 2000 g ($6.8 \times 10^{-2}$ % strain) and −3 for a compression of 2000 g (Fig. 8). The strain sensitivity of a laser-trimmed resistor was considered to be the sum of two terms, one being the intrinsic strain sensitivity related to the conduction mechanism in the material, and the other being the extrinsic strain sensitivity related primarily to thermal expansion coefficient mismatches between resistor/substrate and resistor/encapsulant, etc. Of these two the contribution of former was thought to be small and reversible as observed in untrimmed resistors while the contribution of the latter was thought to be large and irreversible, especially for thick resistors. From scanning electron microprobe quantometer (SEMQ) studies, it was reported that a portion of the alumina from the substrate was dissolved into the resistor film and the amount dissolved was found to be less for thicker films ($> 0.5 \times 10^{-3}$ in ($\sim 1.27 \times 10^{-3}$ cm). This dissolution was believed to reduce the thermal expansion mismatch between resistor and substrate, resulting in low reversible strain sensitivities for thin resistors ($< 0.5 \times 10^{-3}$ in ($\sim 1.27 \times 10^{-3}$ cm)). The difference in behaviour of untrimmed and trimmed resistors was attributed to the additional instability introduced by laser kerf in trimming and the formation of microcracks at the tip of the kerf.

## 8. Environmental tests

To ensure proper functioning of hybrid circuits in various environments, they are subjected to certain specific environmental tests and their quality assessed. The four main environmental factors identified as potential sources of resistor failure, are temperature and humidity, thermal cycling, thermal shock and chemical attack by ambient factors. The presence of humidity or ambient corrosive gases at the resistor surface of the film, despite a conformal coating or hermetic sealing, becomes possible due to the porous nature of the former and defective sealing of the latter.

Coleman [29] studied the long-term stability of Du Pont 1400 resistor systems by carrying out accelerated ageing tests in air and monitoring the resistance changes. Samples of laser trimmed, untrimmed, overglazed, unglazed resistors of DP1411 (10 Ω/□), DP1431 (1 kΩ/□) and DP1461 (1 MΩ/□) compositions were kept in air at 70, 125 and 200° C, respectively, for prolonged hours and percentage change in resistance was monitored. Table VII gives the net percentage change in resistance of 1411, 1431 and 1461 resistors at 70, 125 and 200° C, respectively. Overglazing the resistor or refiring the resistor at overglaze temperature was found to decrease the resistance drift with thermal ageing. The drift in resistance was found to be higher if the ageing was carried out in nitrogen. The aged behaviour of the resistors was attributed to the possible diffusion of ruthenium ions which act as impurity centres and favour tunnelling towards the ruthenate/glass interface. This was thought to reduce the centres available for conduction leading to an increase in resistance. Another ageing mechanism proposed was stress relaxation. The lower change in resistance of the sample refired at 525° C was taken to be evidence for the latter mechanism.

Lovati and Bellardo [19] studied the effect of thermal cycling (−50/+125° C, 20 cycles) and thermal ageing (125, 150 and 175° C; 1 to 1000 h) on the trimmed resistance of Du Pont 1400 series resistors of various geometries. The above tests were carried out after complete saturation of post-trim drift in trimmed resistors. The resistance drift due to thermal cycling was found to be negative for 10 and 100 Ω/□ resistors while it was positive for other resistors of higher sheet resistance. The drift was within 0.4% even for small resistors (0.3 mm × 0.4 mm). However, the resistance drift due to thermal ageing was found to be quite significant (+0.7% after 1000 h) for smaller resistors (0.3 mm × 0.4 mm). For resistors with length > 1 mm, the drift was between 0.05% and 0.2%. Resistors printed with reduced thickness showed lower degree

TABLE VII Change in resistance with time of Du Pont 1400 resistors after ageing in air, after [29]

| Resistor | Change in resistance (%) after 10 000 h ageing in air at | | |
|---|---|---|---|
| | 70° C | 125° C | 200° C |
| DP 1411 (10 Ω/□) | 0.4–0.5 | 2.0–3.0 | 5.0–6.0 |
| DP 1431 (1 kΩ/□) | 0.06–0.07 | 0.3–0.4 | 5.0–6.0 |
| DP 1461 (1 MΩ/□) | 0.09–0.1 | 0.6–0.7 | 4.0–5.0 |

of defectiveness and better stability. Almost similar drifts in resistance due to thermal cycling and ageing were reported by Rapeli *et al.* [20] from their studies on Du Pont 1600 series resistors.

## 9. Power loading or refiring

The primary effect of continuous operation of a circuit under power on the resistor element is the internal heating of the resistor due to power dissipation. The internal temperature of the resistor film due to the above heating depends upon the amount of power handled, area of the film, heat dissipation mechanisms built into the package, power loading of adjacent resistors, etc., and sometimes may be significant enough to cause a permanent drift in resistance. The nature of this drift to some extent can be determined from refiring studies on pure, unglazed resistors. The drift can also be measured directly under load conditions.

Cattaneo *et al.* [14] studied the effect of refiring on electrical and structural properties of Du Pont 1400 Birox resistors. Resistors were processed on two types of substrates, beryllia and alumina, and were subjected to repeated refirings through the peak firing temperature. The percentage change in resistance of Du Pont 1441 (10 kΩ/□) and 1451 (100 kΩ/□) resistors was measured with the number of refirings. The sheet resistance of these resistors was found to decrease significantly (40% to 50% after 10 cycles) with refiring and after a number of firing cycles it reached stable values. $T_{min}$, the temperature where TCR becomes minimum, was found to shift to lower values with refiring. From XRD, SEM and microprobe analysis (MPA) studies on these resistors at different stages of the refiring process, the authors reported that the presence of $RuO_2$ even in a sample fired only once could be attributed to the decomposition of $Bi_2Ru_2O_7$. In addition, one further unidentified phase was detected in XRD patterns of refired samples on the alumina substrate and the concentration of this phase was found to increase with the number of refirings. Because such a phase was absent in refired resistors on the beryllia substrate, this phase was thought to be an aluminate compound resulting from strong interaction between $Bi_2Ru_2O_7$ and the substrate. Based on SEM and MPA studies, the decrease in sheet resistance and shift in $T_{min}$ with refiring, was attributed to a rearrangement of film components with the upper layers of the resistor rich in glass, and the lower layers rich in ruthenate grains.

Pierce *et al.* [7] also studied the effect of refiring on sheet resistance of Du Pont 1400 series resistors. They reported that with refiring the sheet resistance of the resistor would increase and TCR would decrease. This was attributed to the formation of $RuO_2$ which has a lower resistivity and higher TCR value than those of

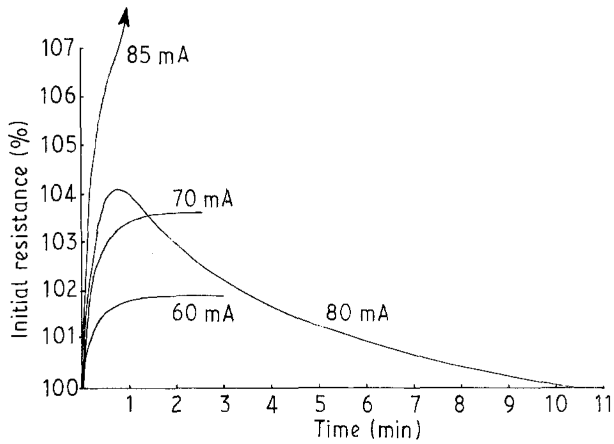


*Figure 10* Effect of increasing load level on Du Pont 1131 resistance, 0.5 mm long, 1 mm wide [30].

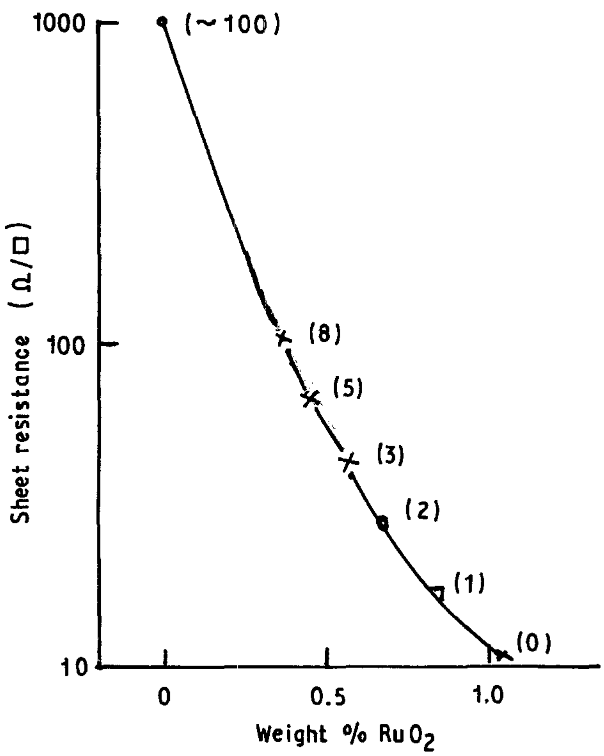


*Figure 9* Variation of $RuO_2$ content against sheet resistance for multiple refires of 10 Ω/□ [7].

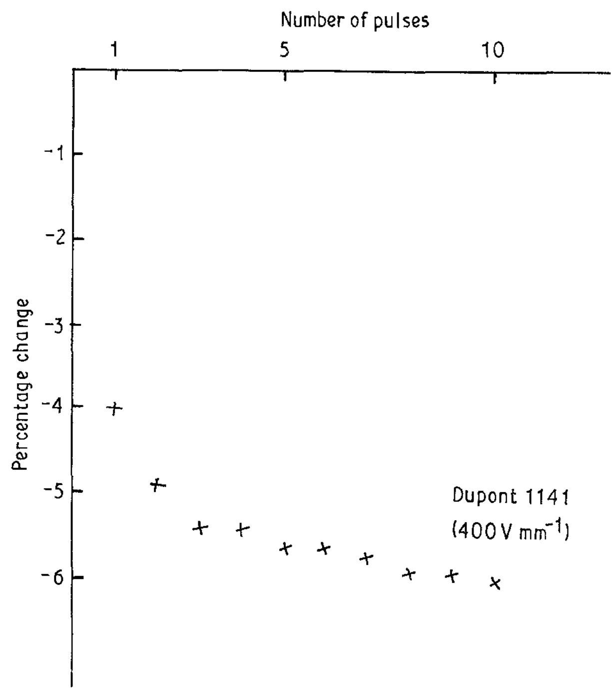


*Figure 11* Effect of repeated high-voltage pulses on Du Pont 1141 resistance [30].

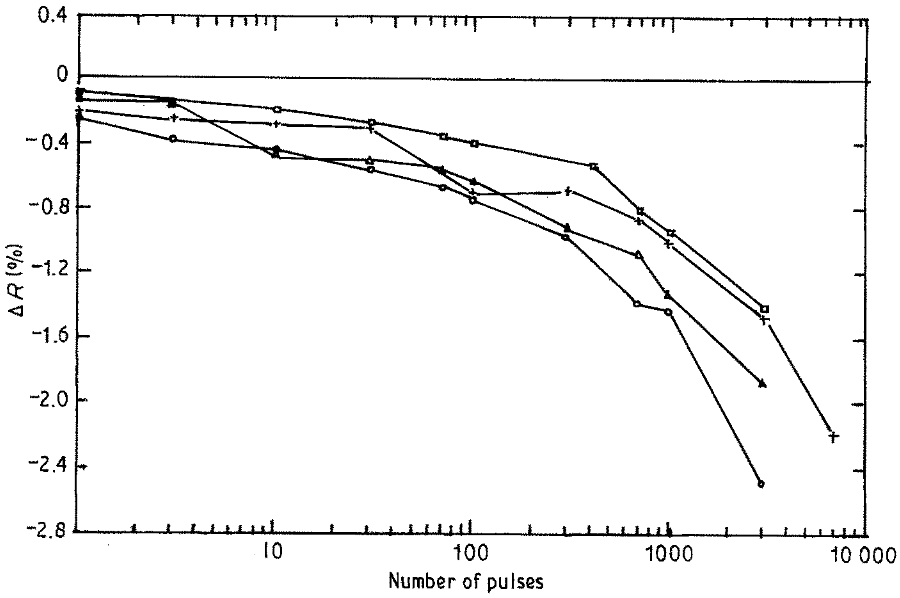


*Figure 12* Effect of number of high-voltage pulses on resistance of 100 kΩ/□ resistor (Du Point 1300 series), $t_c$ = 300 μsec, $V$ = 2.5 kV. Conductors: (○) Pd/Au, (+) Au, (△) Pd/Au, (□) Pt/Au [31].

$Bi_2Ru_2O_7$, during the first firing of the resistor film. During subsequent refirings the $RuO_2$ was found to be converted to $Bi_2Ru_2O_7$ leading to an increase in sheet resistance and decrease in TCR value. Fig. 9 shows variation of sheet resistance with weight percentage of $RuO_2$, the numbers in parentheses indicating the number of refires. McCloghrie *et al.* [30] studied the effects of d.c. loads and high-voltage pulses on Du Pont 1100 series resistors. Figs 10 and 11 show the effect of increasing load level and the effect of repeated high-voltage pulses on resistance changes of 1131 (1 kΩ/□) and 1141 (10 kΩ/□) resistors, respectively. At least four effects were thought to be responsible for the observed changes: (a) temperature coefficient of resistance, (b) voltage gradient coefficient of resistance, (c) thermal degradation due to heat dissipation, (d) high-voltage breakdown effects. Among these, (a) and (b) are reversible effects leading to temporary changes in resistance, while (c) and (d) are permanent effects and often inseparable, leading to permanent changes. These latter were thought to be responsible for the permanent changes in resistance shown in Fig. 10 when load levels were increased from 70 to 80 and 85 mA.

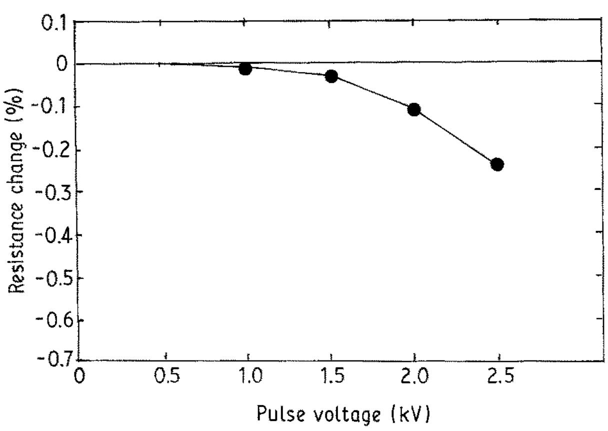


*Figure 13* Effect of pulse voltage on resistance of 100 kΩ/□ resistor (Du Pont 1300 series), $t_c$ = 300 μsec, Pd/Ag electrodes [31].

## 10. High voltages

Design aspects of thick film resistors used for high-voltage applications should receive special attention. The resistor size should be such that the recommended voltage gradient across the resistor should not be exceeded during the operation. The effect of high voltages is more pronounced in high-resistivity materials. The failure mechanisms of these resistors involve microscopic breakdowns within the resistor material or at the conductor/resistor interface or self-heating effects within the material leading to permanent drifts in resistance, TCR and VCR values.

De Lacy [31] studied the performance of Du Pont 1300 series, 100 kΩ/□ resistors under high-voltage pulsing. Figs 12 and 13 show the effects of the number of pulses and pulse voltage on percentage change of resistance of a 100 kΩ/□ resistor, respectively. Resistors were terminated by four different conductor materials (Pd/Ag, Au, Pd/Au and Pt/Au) and the above changes were investigated. Fig. 12 shows that significant changes in resistance occur after about 1000 pulses ($t_c$ 300 μsec, $V$ = 2.5 kV). The conductor/resistor interface was not found to play a major role in the resistance changes. Changes were reported to occur within the bulk of the resistor material. Table VIII gives the average TCR changes after pulsing, which are significant. The changes were attributed to the breakdown of semiconducting parts of the resistor matrix due to high-voltage discharges.

## 11. Conclusions

This brief review shows that to realize fail-safe bismuth

TABLE VIII Average TCR changes for a Du Pont 1300 series 100 kΩ/□ resistor after pulsing [31]

| Termination conductor | TCR (p.p.m.) |
|---|---|
| Pd/Ag | +162 |
| Au | +163 |
| Pd/Au | +198 |
| Pt/Au | +158 |

ruthenate thick film resistors care should be taken in selecting proper materials (conductors, substrates, sealing and encapsulating resins, etc.) and process parameters (furnace atmosphere, cooling rate, fired resistor thickness, etc.). Also, resistors should not be allowed to operate beyond their rated currents, voltages and temperatures.

## Acknowledgements

The authors wish to thank Dr A. Prabhakar, Executive Director, R&D and Mr G. H. Sharma, Dy General Manager, Microelectronics Division for their interest and continuous encouragement.

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*Received 14 February and accepted 19 March 1990*