# Silicon contamination caused by diffusion of silicone into plastic sealed relays



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## Abstract

The most common reason for relay contact failures is the deposition of polluting materials which increase contact resistance or cause corrosion of the contacts. Silicone has long been known to be a source for significant problems when used in the environment of electrical contacts. Therefore, to curb contamination, relays now use plastic covers sealed with epoxy glues. Nevertheless, we still find sealed relays in various applications with increased contact resistance due to silicon contamination. Obviously, relay covers can, under certain thermal conditions, exhibit a “breathing” phenomenon which can cause contaminants to accumulate within the protective cover. To ascertain typical parameters responsible for this, we tested sealed signal relays in a silicone environment at different temperatures. Moreover different loads and switching cycles were applied to the contacts in order to determine the worst conditions. FTIR-spectroscopy as well as colorimetric measurement methods were performed to quantify the compounds. In addition, the contact quality after exposure was analysed by a SEM / EDX microscope. The investigation showed that low molecular weight of silicone oil diffuse into a sealed relay under certain conditions. A correlation between switching load and the polymerisation of a harmful layer on the contacts could be defined.

## 1. Introduction

Failures in electromechanical relays associated with chemical contamination on the contact surface can be traced to a variety of causes. Contamination may arise from manufacturing processes that create dust and particles or outgassing from plastic parts. Especially with sealed relays it is highly probable that the manufacturer is responsible for this kind of failure.

In the past years we have observed frequent common customer claims resulting from chemical contamination. The most peculiar claims result from silicon-oxide ($SiO_2$) on the contact surface. In our factory all components used in the manufacturing process are checked for organic silicone, the basic material for $SiO_2$ layers. Therefore we can exclude silicone contamination in the manufacturing process. Thus we assumed that organic silicone diffused into the sealed relays.

To be able to investigate this process in detail, one must understand the environment in which the relay operates and its electrical parameters. In many cases the contaminant could be located directly on the PC board. Some silicon sources are coating materials and various glues used to fix parts on the board. This was the reason why we started intensive investigation of the diffusion process of silicone into sealed relays. The reason for the increase in contact resistance is the formation of $SiO_2$ on the contact surface. This process starts during the switching process due to the decomposition of silicone vapour under high temperature. The cracking temperature of the silicone molecules is easily reached if there is a breaking arc while switching the relay. This is given when the voltage is above 12V (depends on contact material and current). $SiO_2$ is a typical insulator and on affected contacts a fine crystalline structure is built up within a few thousand operations, depending on electrical load and the amount of organic silicone in the vapour phase. If this is the case, a sudden increase in contact resistance can be observed. Especially in small signal relays the contact force is too low to remove these very obstinate layers from the contact surface [1][2].

In the present study we show that there is a significant dependency for contact failures in a silicon environment, even in relays sealed with epoxy glue. Using various analytical methods we were able to find a correlation between the silicon compound we used in the environment and detected silicon compounds we found inside the relay after the test.

# 2. The experiment

## 2.1 Description of the relay

The sealed micro relays used for the diffusion test are from various production lines in Europe and Asia. We used standard relays without special treatment. The size was 20 x 9.9 x 9.8 mm. The relay consists off two changeover twin contacts. The contact surface is a 5 µm thick Au-layer with a contact force of approx 12 cN. All relays were checked 100% for contact resistance and tightness before and after the test. The cover material is 0.7 mm thick PBT and the moulding material is epoxy resin.

## 2.2 Analysis of silicone oil

Initially we analysed several industrial oils to see if they contain organic silicone. To identify the amount of organic silicone within these samples we used a chemical detection method. In this "silicon colorimetric analysis" silicon atoms are ionised through chemical treatment. In a subsequent reaction in a molybdenum alkaline solution the colour changes to blue. Since the depth of the blue is proportional to the amount of silicon, it is possible to obtain a quantitative analysis to a level of 0.5 ppm by optical photometer spectroscopy. One of the best results we obtained was with a standard motor oil. Here we found a value of more than 10 ppm organic silicon. This oil is used for all following measurements.

## 2.3 Test with open micro-relays

Responsible for the building of $SiO_2$ layers is the internal molecular structure of the silicone oil [6]. In the beginning we investigated the influence of the oil on switching contacts. Therefore we tested open micro-relays in a silicon oil environment. The cover of the relay was removed so that the relay contacts were directly exposed to the silicon vapour. For this switching test we used a hermetically sealed, two-litre test chamber, in which we put 50 ml of silicon oil. Under the following conditions: switching rate 0.1 Hz (duty cycle 50%) 12V DC and three different currents: 1.0 A, 0.5 A, and 0.12A (5 relays each). To speed up the process the temperature in the test chamber was increased to 60°C. The contact resistance was detected online via a transient recorder by four terminal potential drop with a measuring current of 10 mA. These are very similar conditions to those reported by Tamai [3][4]. To get a rough estimation about the concentration of silicone, we could compare his results with our present measurements. A chemical reaction is expected after a few thousand operations with a change in contact resistance from around 20 mOhms to a few kilo-Ohms. After the test all affected contacts were examined by optical microscope and scanning electron microscope (SEM) equipped with an energy dispersive x-ray (EDX) analysis system.

## 2.4 Test with sealed micro-relays

To simulate typical environmental conditions our customers deal with in their applications, we prepared 50 relays in the same test chamber described in section 2.2.. The electrical load was fixed to 12V and 1A. To speed up the chemical reaction the temperature was switched from 20°C up to 60°C in one-hour cycles. This caused a "breathing effect", which increased the diffusion rate into the relay. Furthermore the time between the electrical switching cycles was set to 1 second on/ 2 minutes off. The adsorption of silicon on the contact surface can reform itself in the time between. This fact was reported in a publication from Ishino and Mitami [5].

After the test all affected relays were opened and the contacts were examined by microscopic inspection. All inner parts of the relay were treated with Freon to collect all the possible silicone that had diffused inside. The solvent was subjected to infrared spectroscopy (FT-IR) and compared with the silicone vapour outside. This method is extremely well suited for microgram quantities of organic material.

To avoid systematical failures we prepared a second PC board in the same environment without silicone oil. For this control group (same lot number as samples used for the silicone test) electrical load, temperature and switching frequency were same.

# 3. Results and Discussion

## 3.1. Open micro relays

The effect of the silicon vapour on the contact resistance of the open relays is shown in Figure 1 for three typical samples. All the switching contacts were more or less affected. For each current the value of the contact resistance is plotted against the number of operations. The higher the current the faster the increase in contact resistance. In Figure 2 the corresponding optical micrographs are shown. For each load the contact area shows a very significant pattern of brown to black residues on the contact surface. Further investigation by EDX shows a significant amount of carbon and silicon in the switching area of the contact. The ratio of silicon increases slightly when the current is increased.

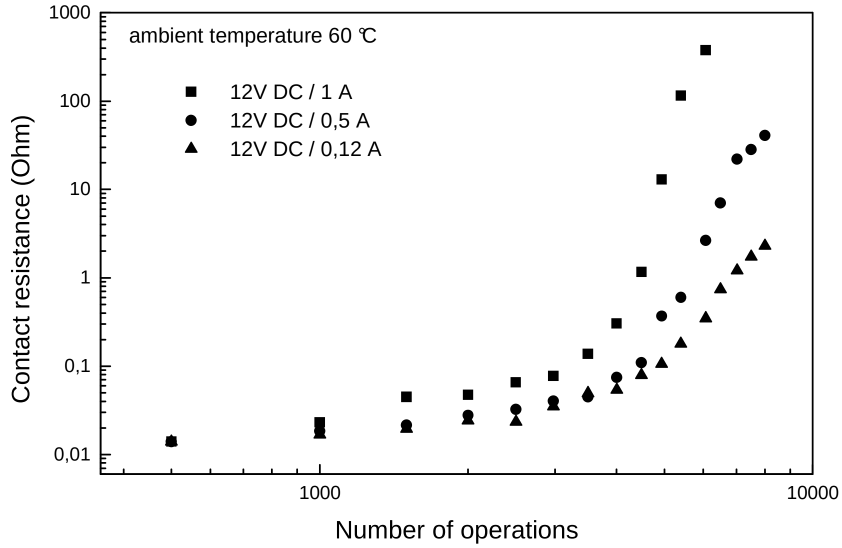
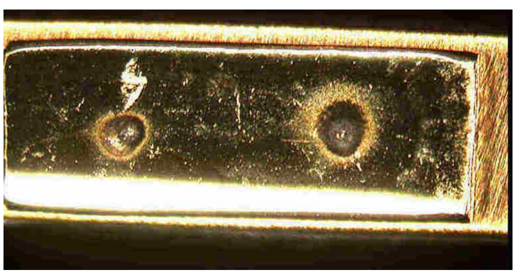
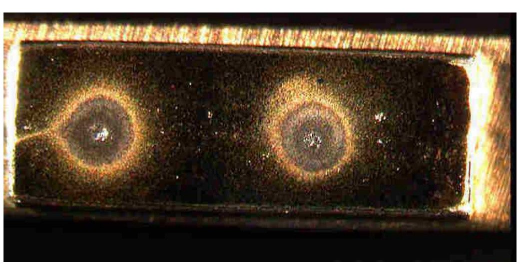



**Fig.1: Increase of contact resistance depending from number of operations for three different loads**

**a) Load 12V DC / 0,12 A**



**b) Load 12V DC / 0,5 A**



c) **Load 12V / 1 A**

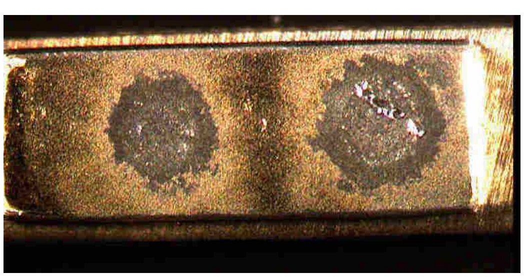


**Fig.2: Picture of contacts for different loads**

Finally we could show that in a typical industrial oil there is enough silicone to damage the contact surface of a small micro relay. The increase in contact resistance is caused by silicon and carbon layers on the contact surface. Especially Si building up a crystalline structure of $SiO_{2,}$ which leads to an infinite contact resistance.

When we compare these results with the publication of Tamai [3][4] we can estimate an amount of ~100 ppm of active silicon molecules in our test environment.

## 3.2 Sealed relays

In the test with the sealed relays we found the first slight increase in contact resistance after 50 000 operations. Due to the slow switching cycle with more than 2 minutes off, it takes about one month to obtain a small measurable deviation compared to the control group. But after another 50 000 operations we could demonstrate a very clear result. These data are shown in Fig. 3. The circles show the measured contact resistance for each relay preceding the test. The squares show the contact resistance of the relay after 100, 000 operations and two months in a silicon environment. To simplify representation the data are sorted in ascending order. As an initial result we can see that only around 20% of the relays show an increase of contact resistance for more than 1.0 Ohm. After opening these relays and inspecting them microscopically, we found a very similar result compared to the uncovered relays. Fig. 4 shows a SEM picture of the contact area with the typical $SiO_2$ polycrystalline film and high amount of carbon covering the whole switching area. The elements were identified by EDX. This measurement is shown in Figure 5. Beyond the elements silicon and

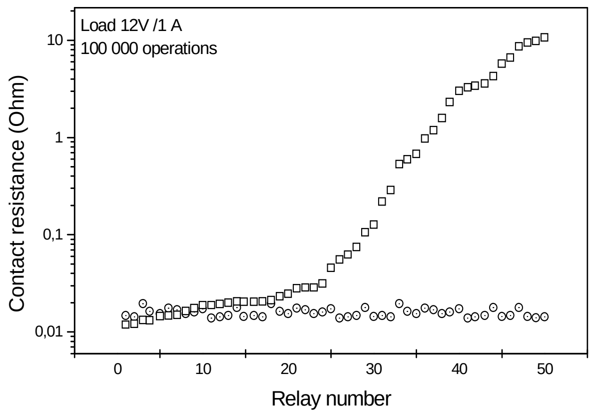



**Fig. 3 : Contact resistance of 50 relays initially and after 100 000 operations at 12V/1A**

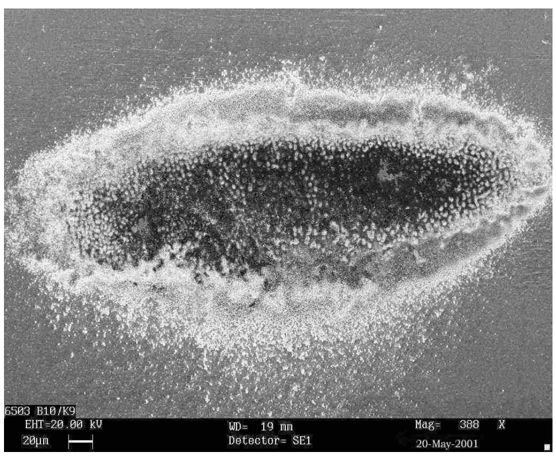



**Fig. 4 : SEM picture from contact area**

carbon we could also detect oxygen as a foreign material. The gold and silver peaks are background signals from the contact material.

After opening the relays all inner parts were cleaned ultrasonically with Freon. Afterwards the solution was analysed by FT-IR. In Fig. 6 we show the spectrum in comparison with the spectrum we received from our silicone oil. The correspondence between the two measurements is obvious: There is a very clear indication that we have diffusion from the outside to the inside of the relays.

If we assume diffusion occurs through the relay-cover, the amount of organic silicon inside the relay should be the same for all tested relays. Also the increase in the contact resistance should be comparable. The surface area and the thickness of the cover, which is responsible for the diffusion rate, could not explain such a big deviation.

A possible explanation for the low reproducibility in the increase of contact resistance is the sealing process. We estimate that most of the silicone finds its way inside the relay between the cover, and the body block or along the pin. This hypothesis has not yet been proven, but in these areas the building up of microchannels due to thermal stress is most likely.

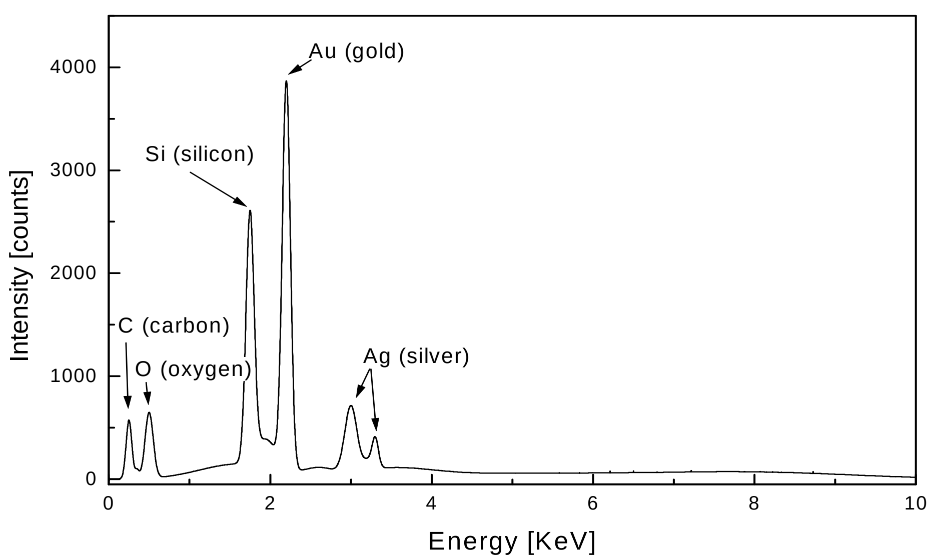



**Fig. 5: EDX spectrum of the contact area given in Figure 4**

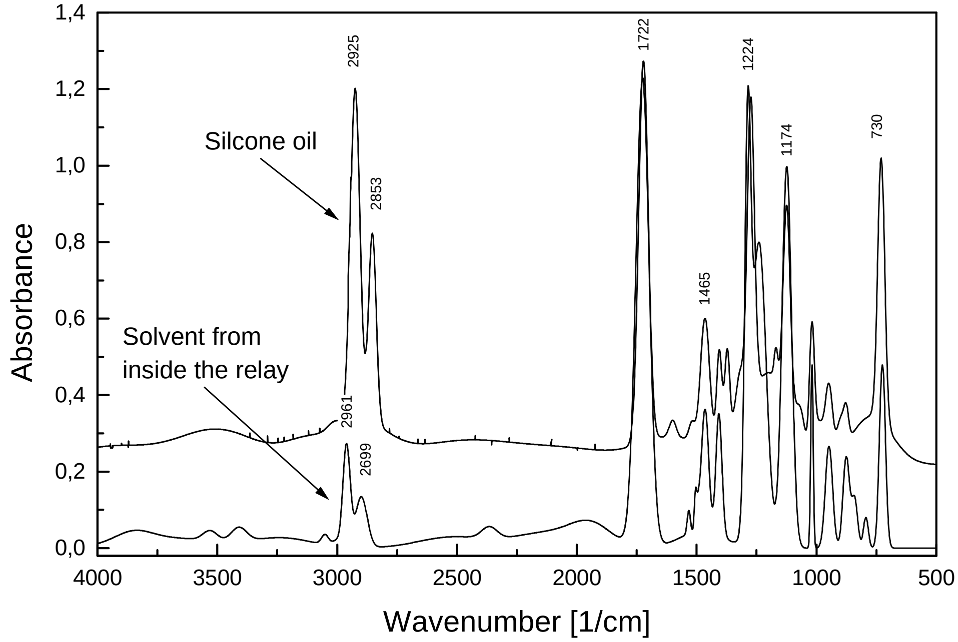



**Fig. 6 : FTIRspectrum (comparison of pure silicon oil and the solvent from inside the relay)**

## 4. Conclusion

Diffusion of contaminants e.g. silicone in to a sealed relay is possible under strenuous ambient conditions. This results in the formation of a tenacious, highly resistive film containing silicon.

## 5. Literature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