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# CHARACTERISATION OF OUTGASSING CONTACT MATERIALS FOR MINIATURE RELAYS

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**Abstract:** Systematic investigations of vacuum outgassing for hermetic relays with AgCdO/AGPHOR and PdAg30 Au electroplated contacts were done. The experimental vacuum system constructed and built at our institute was additionally equipped with quadrupole mass spectrometer for residual gas analysis. Prior to the final encapsulation the relays were baked at the constant temperature of 125°C and outgassed in vacuum $1x10^{-5}$ mbar for several hours. The residual atmosphere was analysed every hour; in residual gas mixture, hydrogen, carbon monoxide, carbon dioxide, water vapour and remanents of cleaning chemical agents (ethanol, trichlorethylene) and hydrocarbons were found. Residual gas analysis showed that the concentration of impurity gases depends on materials and their treatments in different technological phases.

# Karakterizacija razplinjenega kontaktnega materiala za miniaturne releje

**Ključne besede:** releji miniaturni, releji elektromagnetni, releji hermetični, deli sestavni, razplinjevanje vakuumsko, lastnosti kontaktov, pozlatitev elektrokemijska, upornost kontaktna, elektronika profesionalna, preskušanje, rezultati eksperimentalni, AES Auger spektroskopija elektronska

**Povzetek**: Sistematično smo raziskovali hermetične releje z AgCdO (AGPHOR lot folija) in PdAg30 elektrokemijsko pozlačenimi kontakti. Eksperimentalni vakuumski sistem, ki smo ga uporabljali, je bil konstruiran in narejen na našem inštitutu. Dodatno smo ga opremili s kvadrupolnim masnim spektrometrom za analizo preostale atmosfere v relejih. Pred dokončno inkapsulacijo smo preiskovane releje razplinjevali nekaj ur pri konstantni temperaturi 125°C v vakuumu $1x10^{-5}$ mbar. Vsako uro smo analizirali atmosfero v relejih. V izmerjeni mešanici plinov smo odkrili vodik, ogljikov monoksid, ogljikov dioksid, vodno paro, ostanke kemijskih čistil (etanola in trikloretilena) in klorovodike. Analiza je pokazala, da je koncentracija plinskih nečistoč odvisna od materialov samih pa tudi od njihove obdelave v različnih tehnoloških postopkih.

## 1. INTRODUCTION

Contamination film formed on the surface of electric contacts is one of the most serious causes of failure of hermetic relays. It deteriorates the contact resistance and device reliability. The most common types of contamination films are oxides and other corrosion products particles, layers formed by thermal diffusion processes, debris produced by mechanical wear and fretting, further evaporation outgassing, and condensation on contact surfaces of volatiles from isolation materials and those originating from manufacturing processes (1-9). Systematic investigations of vacuum outgassing of hermetic relays are done. The experimental vacuum setup was designed and assembled at Institute for Electronics and Vacuum Technique, Ljubljana. It has been additionally equipped with quadrupole mass spectrometer for residual gas analysis.

## 2. EXPERIMENTAL

The experimental set up, designed and assembled specifically for vacuum outgassing of hermetic relays is shown in Fig. 1. The vacuum system consists of rotary

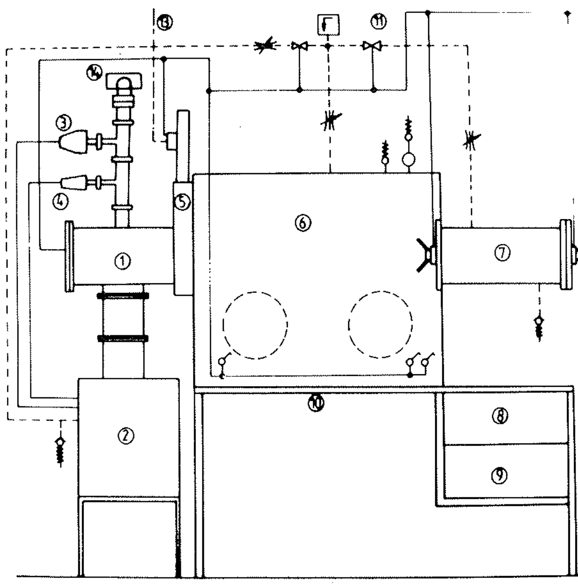



*Fig. 1a: Experimental vacuum system (scheme) for outgassing process for hermetic relays: 1-vacuum chamber, 2-vacuum system, 3-PNG head, 5-mass spectrometer, 6-plate valve, 7-$N_2$ chamber for final hermetic relays incapsulation*

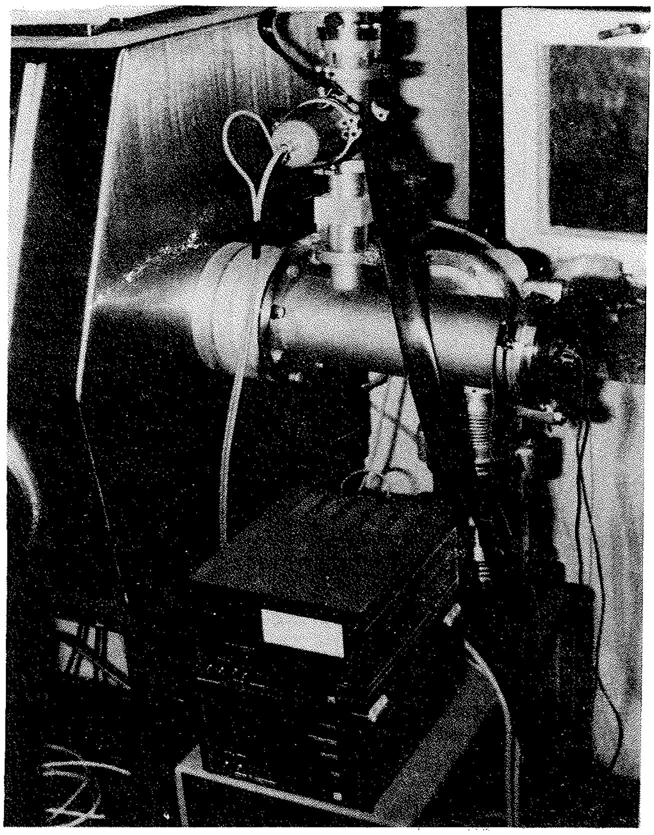


*Fig. 1b:* *Experimental vacuum system (photograph)*

vane pump and turbomolecular pump enabling to attain a vacuum of at least $1x10^{-6}$ mbar. The vacuum chamber which can be baked up to 300°C is equipped with a resistive heater. A thermocouple Fe-CuNi, 0.5 mm in diameter, is in thermal contact with one of the outgassed relays in the middle of the vacuum chamber. For residual gas analysis the quadrupole mass spectrometer Leisk 1000 M with AMU 2-100 has been used. Prior to the final hermetic incapsulation the relays are baked at constant temperature of 125°C in a vacuum of $1x10^{-5}$ mbar for several hours. The residual atmosphere has been analysed every hour.

## 3. RESULTS AND DISCUSSION

The first analysis of residual atmosphere was made in an empty and unbaked stainless steel chamber after a few days of continuous pumping. The base pressure in chamber was $2x10^{-6}$ mbar. Mass spectrum in Fig. 2 shows the outgassing products in the empty chamber. This spectrum is typical for a standard vacuum system with very small air leaks and no unusual gas sources; it shows that the most prominent outgassing product in the empty vacuum chamber is a mixture of water vapour, CO and $N_2$; small quantities of hydrogen and some hydrocarbons are also released in the empty chamber at this temperature as expected.

The spectrum in Fig. 3 was obtained after one hour's baking of the empty chamber at 125°C. This spectrum

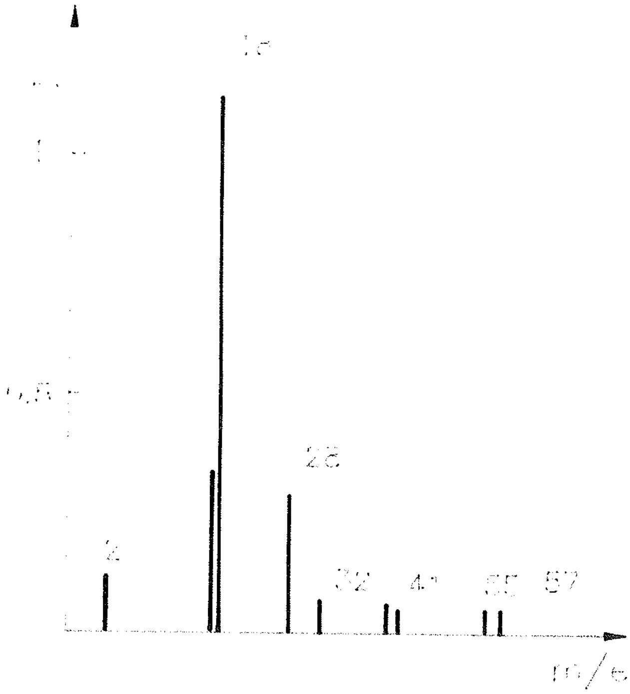


*Fig. 2:* *Mass spectrum showing the outgassing products of the empty chamber*

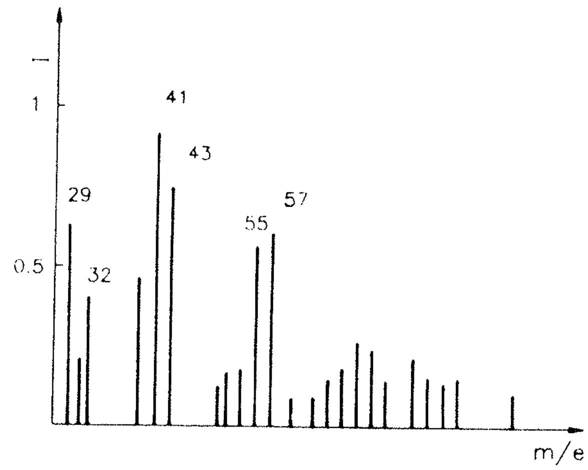



*Fig. 3:* *Mass spectrum of the outgassing products of the empty chamber after one hour baking*

shows the main outgassing product consisting of water vapour, CO and $N_2$ and unusual peaks typical for ethanol, trichlorethylene (mass 31, 45, 60) supposedly remanents from cleaning processes; some hydrocarbons are also released.

The spectrum in Fig. 4 was obtained after 24 hours baking of the empty chamber at the same temperature. Total pressure attained was $1.3x10^{-6}$ mbar. This spectrum is similar to that shown in Fig. 2, which shows the empty chamber without baking.

Finally, the mass spectrum represented in Fig. 5 shows the outgassing products of miniature relays baked at 125°C. The system pressure in this case was $1x10^{-5}$ mbar. The spectrum was registered 30 minutes after bringing the miniature relays up to the temperature. In the spectrum considerable partial pressures of ethanol,

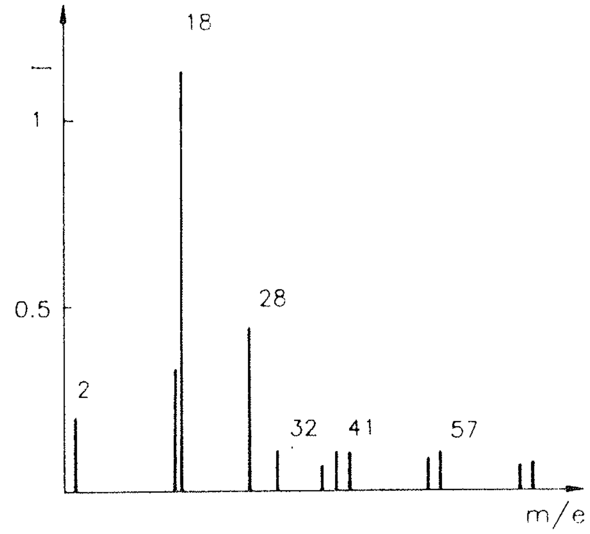



*Fig. 4:* *Mass spectrum of the outgassing products of the empty chamber after 24 hours baking*

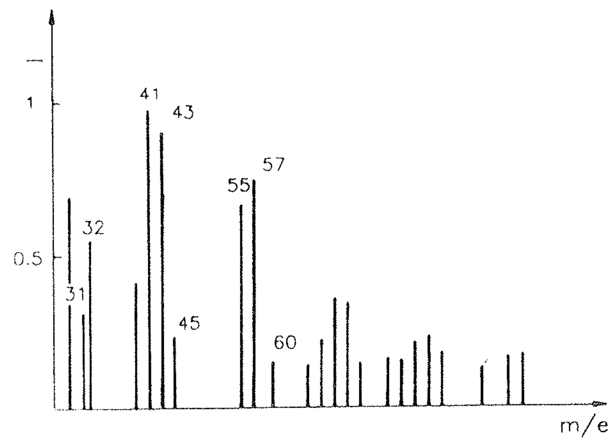



*Fig. 5:* *Mass spectrum of the outgassing products of miniature relays (contacts PdAg30/Au) at 125°C in a vacuum of $1x10^{-5}$ mbar after 30 minutes*

hydrocarbons, oxygen and $CO_2$ can be seen. Other spectra indicate also appreciable concentrations of water vapour, CO and $N_2$.

Residual atmosphere was analysed every hour. After 24 hours of relay outgassing process the spectrum in Fig. 6 was registered. The background system pressure was $3x10^{-6}$ mbar. A comparison of this spectrum with that shown in Fig. 4 for the empty vacuum chamber at 125°C, reveals that both spectra are quite similar, indicating that the baked relays are ready for final incapsulation. The most useful and often the simplest method for detecting contamination on an electric contact surface is to determine its contact resistance.

Contact resistance of vacuum outgassed relays with PdAg30/Au contacts was measured immediately after hermetic incapsulation and later in constant time intervals of 7, 14, 21 and 42 days. For a comparison the contact resistance of nonoutgassed relays was measured. The results are collected in Table 1. Initial contact resistances of nonoutgassed and outgassed relays were very similar. However, the difference in

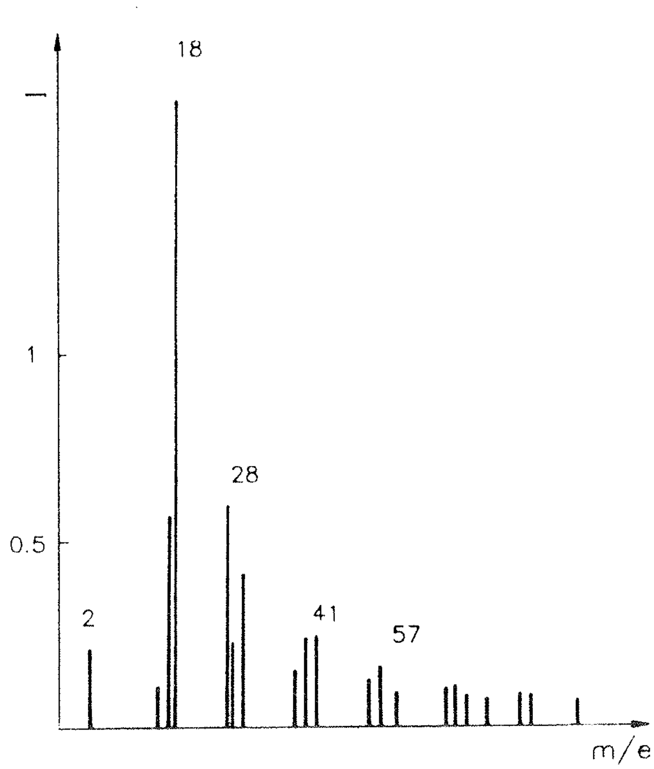



*Fig. 6:* *Mass spectrum registered after 24 hours of the relay (contacts PdAg30/Au) outgassing process*

| Sample no. nonoutgassed relays | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| R (mΩ) after incapsulation | 65 | 70 | 55 | 60 | 70 | 55 | 65 | 70 | 60 | 55 |
| R (mΩ) after 7 days | 120 | 130 | 110 | 140 | 100 | 90 | 120 | 145 | 100 | 90 |
| R (mΩ) after 14 days | 205 | 180 | 175 | 250 | 200 | 150 | 170 | 220 | 205 | 195 |
| R (mΩ) after 21 days | 230 | 190 | 175 | 255 | 205 | 160 | 170 | 230 | 210 | 205 |
| R (mΩ) after 42 days | 250 | 300 | 210 | 280 | 250 | 210 | 180 | 245 | 230 | 220 |

| Sample no. outgassed relays | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 |
|---|---|---|---|---|---|---|---|---|---|---|
| R (mΩ) after incapsulation | 70 | 60 | 55 | 75 | 70 | 60 | 60 | 50 | 60 | 65 |
| R (mΩ) after 7 days | 70 | 60 | 55 | 80 | 70 | 60 | 60 | 50 | 60 | 65 |
| R (mΩ) after 14 days | 70 | 60 | 55 | 85 | 70 | 60 | 65 | 50 | 60 | 65 |
| R (mΩ) after 21 days | 70 | 60 | 55 | 85 | 70 | 60 | 65 | 50 | 60 | 65 |
| R (mΩ) after 42 days | 70 | 60 | 60 | 85 | 70 | 60 | 65 | 50 | 65 | 65 |

*Table 1:* *Contact resistance of nonoutgassed and vacuum outgassed relays (contacts PdAg30/Au) measured immediately after hermetic incapsulation and in constant time intervals after 7, 14, 21 and 42 days*

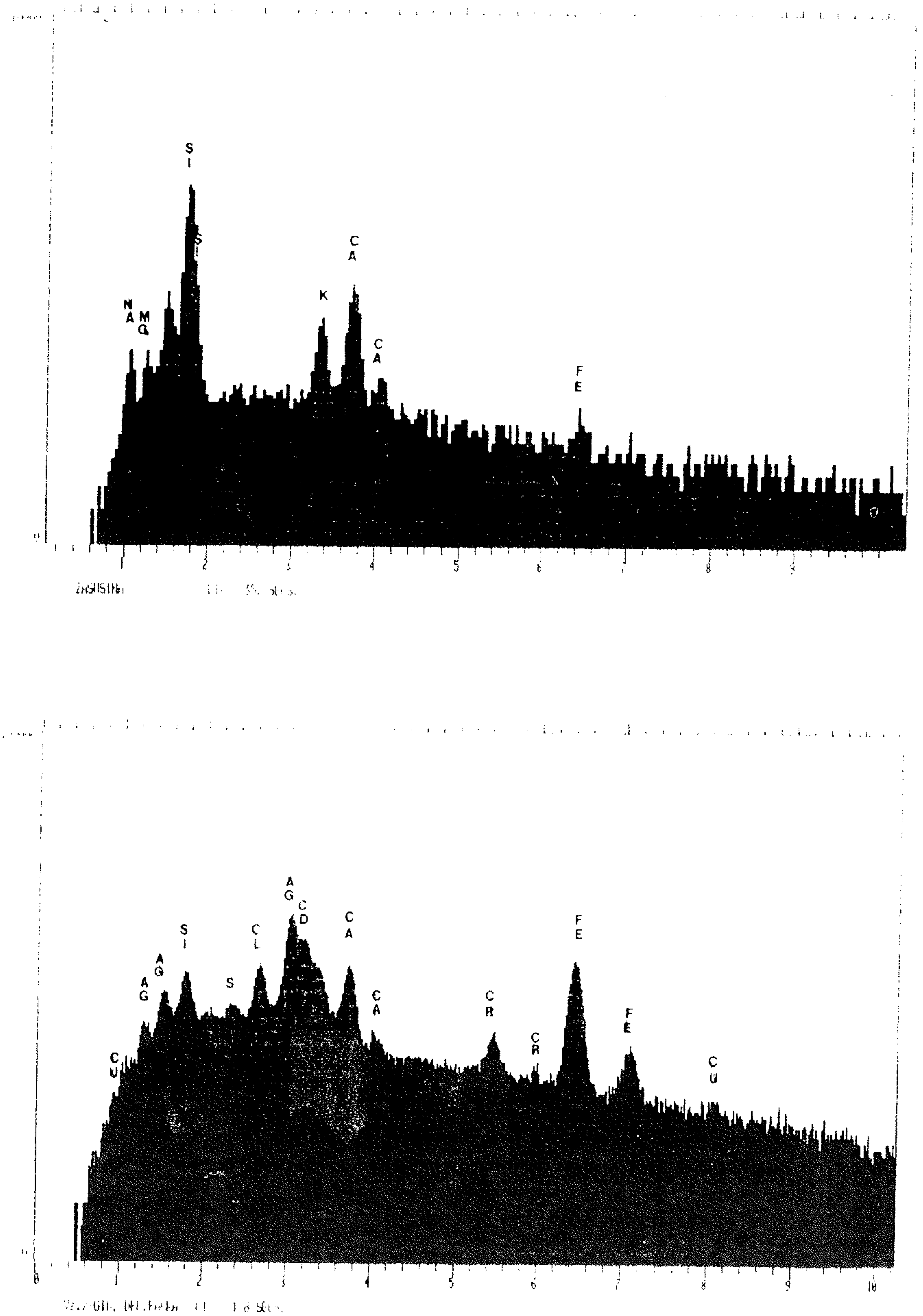
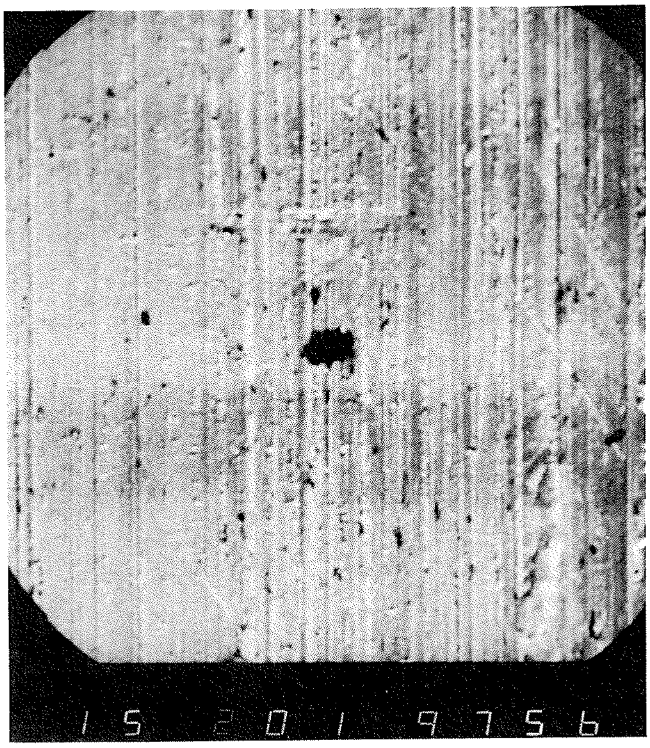



*Fig. 7a:* *Mass spectra of the outgassing products of contaminated miniature relay (contacts AgCdO/AGPHOR) at 125°C in a vacuum of $1x10^{-5}$ mbar after 24 hours*



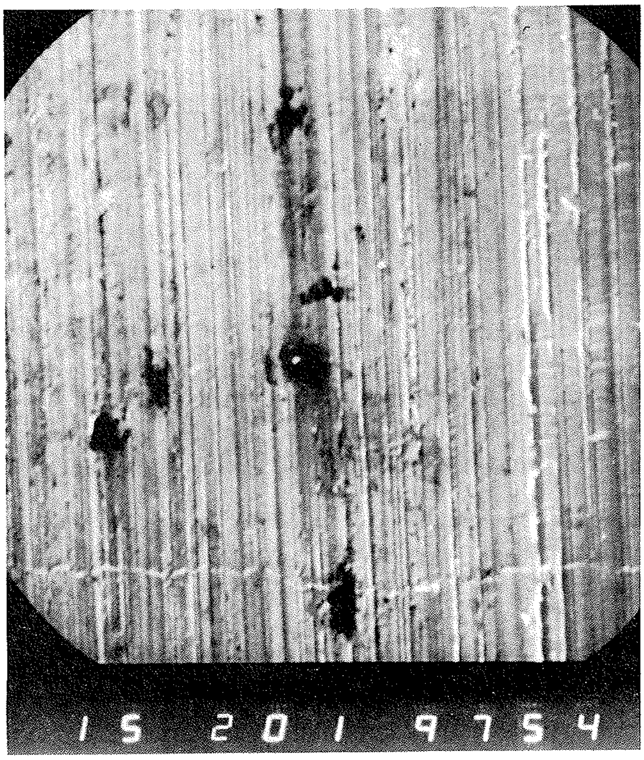




*Fig. 7b: Pictures of the contaminated contact after outgassing*

contact resistance was as early as after 7 days enormous. In relays which were not treated with vacuum outgassing contact resistance increased higher than it is qualified in current standards for miniature hermetic relays (0.1 ohm max).

When contact resistance of miniature relays with AgCdO contacts was measured after outgassing considerable increase was noticed. Therefore some relays were opened and their contacts analysed (Fig. 7a and 7b). Dark spots found on the contacts probably caused the increase of contact resistance. It seems that the relays mentioned were not sufficiently outgassed. Detailed examination of the phenomenon is planed in the near future.

## 4. CONCLUSIONS

The experimental vacuum system set up was designed and assembled specifically for vacuum outgassing of hermetic miniature relays. Systematic investigation of vacuum outgassing of hermetic relays were done. Residual gas analysis shows that outgassing products consist of remanents of cleaning chemical agents (ethanol, trichlorethylene), a mixture of water vapour, CO, $N_2$ and some hydrocarbons originating from rotary pump oil.

The results of experiments described above show clearly that the outgassing process is indispensable and should be included in the technology for producing of reliable hermetic relays qualified to MIL-R-3906 and MIL-R- 5757 standards.

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