

Influence of Temperature and Humidity on Titanium Electrodes Intended for an Above Normative Conditions Sensors

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INTRODUCTION

A series of test titanium electrodes, intended for a new generation of hydrogen sensors was prepared. This new generation of hydrogen sensors is expected to operate in above normative conditions. In order to investigate the influence of temperature and moisture on titanium electrodes a series of experiments was conducted. These test allowed to measure resistivity as a function of temperature and to determine the long-term stability of electrical parameters of electrodes. Results will be used in fabrication of electrodes for a new generation of hydrogen sensors.

TEST SAMPLES

For the investigation a series of test samples were prepared. Test electrodes were manufactured in a form of 2 mm wide and 30 mm long titanium stripes on alumina substrates. Titanium metal films were deposited using the electron beam bombardment vacuum evaporation method. Samples were divided into three groups. First two groups were used to conduct initial short term test. The samples from the last group were used in a long term test of stability.

SHORT TERM TEST OF STABILITY

Group I samples were used to determine, what is the influence of moisture on titanium electrodes. The test was performed in temperature of 23°C and relative humidity of 80%. Duration of the test was 14 hours. Resistance slightly increased at the beginning of the test and remained at the level of 101,3% of initial value [Fig. 1].

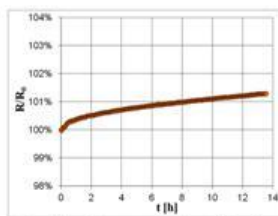


Fig. 1. Resistance as a function of time for $T = 23^{\circ}\text{C}$ and $\text{RH} = 80\%$.

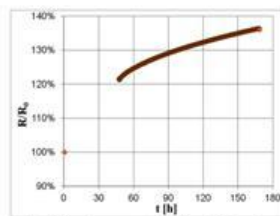


Fig. 2. Resistance as a function of time for $T = 80^{\circ}\text{C}$, $\text{RH} = 80\%$ (R_0 for $T = 80^{\circ}\text{C}$).

Group II samples, exposed to moist air ($\text{RH} = 80\%$) of temperature of 80°C, shown an increase of resistance during the test [Fig. 2]. It seem that, if given enough time, the resistance would reach a stable value.

LONG TERM TEST OF STABILITY

During the long term test of stability (samples from Group III) temperature changes were done in following phases:

1. "annealing" - During this phase temperature was constant and of value of 165°C.
2. "heating" - After the end of phase 1, temperature was lowered down to -35°C. Then it was increased to 165°C at the rate of about 27°C per hour.
3. "cooling" - After the temperature reached 165°C it was decreased back to -35°C at the same rate (27°C per hour).

Total time of all three phases was about 24 hours. Such thermal cycle was repeated six times [Fig. 3].

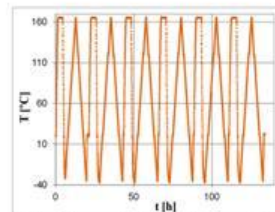


Fig. 3. Temperature during long-term test.

Results of measurements for Group III samples show, similarly to Group II, that resistance initially increased and would have reached a stable value in time [Fig. 4]. It can be seen that after each heating and cooling cycle [Fig. 5] resistance does not return to its initial value - after each cycle it is higher. It can also be seen that in each heating and cooling cycle the width of the loop decreases.

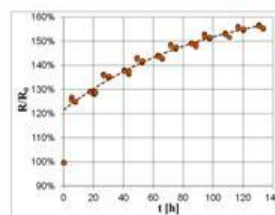


Fig. 4. Resistance measured at $T = 20^{\circ}\text{C}$ as a function of time.

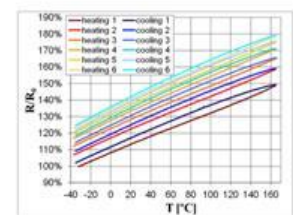


Fig. 5. Resistance as a function of temperature.

CHEMICAL MICROANALYSIS

The results of chemical microanalysis are presented in Fig. 6. Reference sample and two samples annealed at 80°C and 160°C were measured. The increasing of temperature causes the increasing of aluminum and oxygen signals, moreover at 160°C silicon peak is visible. It seems that densification of deposited layer appear, decreasing thickness.

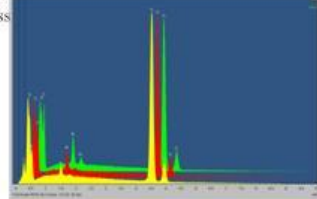


Fig. 6. Results of chemical microanalysis for reference sample (yellow) and samples annealed at 80°C (red) and 160°C (green).

CONCLUSIONS

1. No significant influence of moisture at room temperature was observed.
2. Annealing at temperatures of 80°C and 165°C result increase of resistance.
3. It can be estimated that annealing at temperature of 165°C for 250 hours would be enough to stabilize the resistance of electrodes.

ACKNOWLEDGEMENTS

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