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Screen-printable type S thermocouple for thick-film technology

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Abstract

The aim of this work was to evaluate a novel screen-printable PtRh thick-film paste as a part of a Pt-PtRh thermocouple (type S) that can be fully manufactured in standard thick-film technology. The newly developed PtRh thick-film paste (90% Pt, 10% Rh) was used together with a typical platinum paste. Thus, a new thick-film type S thermocouple was developed. The temperature sensor characteristics agree very well with a type S wire thermocouple. The paper reports on the sensor preparation, characterization tests, and on first very promising aging tests.

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Keywords:

1. Introduction

The application of thermocouples as temperature sensors is well known and already established, a thermocouple itself usually consists of two connected wires with different Seebeck coefficients, *S*. For classical thick-film techniques using screen-printing and firing, no standardized solutions exist. Scattered attempts apply non-standardized Au and Pt inks [1, 2, 3, 4], but on a formulation that leads reliably to long-term stable devices with a characteristics according to IEC or ANSI has not been reported. The difficulty in preparation of standard thermocouples as thick-film structures

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is the availability of appropriate thermocouple materials. Typical standard thermocouples base on Nickel and Nickel alloys (types E, J, K, M, N, and T). For high temperatures, Pt/PtRh alloys serve as materials for thermocouples (types B, S, and R). Nickel-based thermocouples are very popular (especially types J and K), but they can hardly be realized in standard thick-film process due to the oxidation of Nickel and Copper at temperature above 450 °C. The here-presented newly developed PtRh thick-film composition (90% Pt,10% Rh) allows to construct thick-film type S thermocouples. They can be fired in air atmosphere, starting from 950 °C and therefore can be easily integrated into existing thick-film components and devices. Firing at higher temperatures is also possible, but was not studied here.

2. Preparation of thermocouples

The screen-printed structure on alumina substrate is shown in Fig. 1. It consists of a thermocouple with a length of about 45 mm. The printed line width is 150 μm . The outer arm is made of platinum (DuPont 9141R) and the inner arm of the evaluated PtRh ink, respectively.

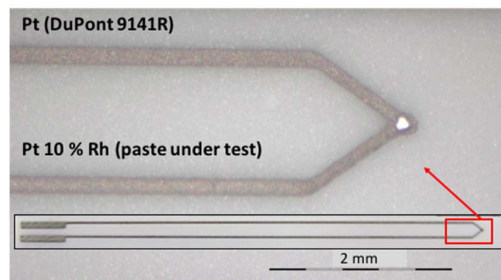


Fig. 1. Fired type S thermocouple

The structures were screen-printed on 96% alumina substrates (CeramTec 708S) using 325 mesh stainless steel screens with a film thickness of 20 microns. The printing speed was 9 cm/sec. After leveling for 10 minutes, the structures were dried at 135 °C for 15 minutes and after that separately fired in a quartz tube furnace at 950 °C (35 min. heating, 15 min. peak, and 35 min. cooling). For extended measurements (not shown in this study), on the reverse side of the substrate, a platinum heater was deposited for heating purposes. The heater was finally covered with an insulation layer (QM42, DuPont) fired at 850 °C. Fig. 1 shows the fired thick-film thermocouple.

3. Evaluation of the structures

The functionality tests were conducted in a tubular furnace with controlled temperature. A type S wire thermocouple was placed very close to the screen-printed one (Fig. 2).

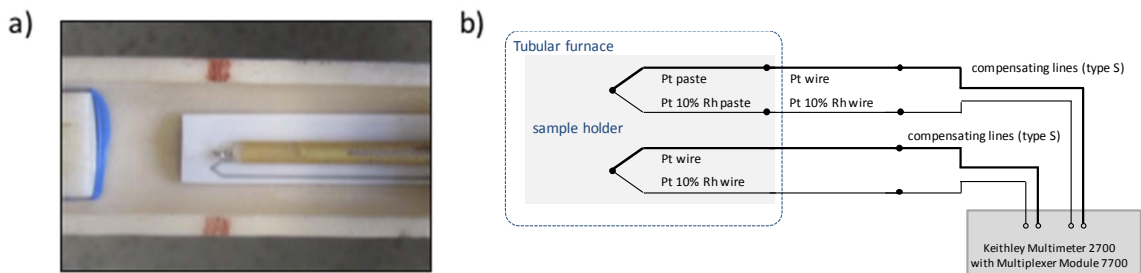


Fig. 2. Evaluation setup: (a) sample holder with the arrangement of the thermocouples, (b) setup (wiring).

In the first tests, we compared both thermocouples by heating the furnace stepwise to 800 °C. First, the thermovoltage of thick-film thermocouple was recorded. From the measured thermovoltage, a temperature was calculated according standard of National Institute of Standards and Technology (NIST) [5]. In that way we are able to confirm that the output voltages of thick-film thermocouple complies with the German and European standard DIN EN 60584. In the further measurement, the thermovoltages of both thermocouples were transformed to a temperature by the built-in conversion function of the Keithley 2700 Multimeter (operated with a Multiplexer Module 7700). The results are shown in Fig. 3. The thermocouples respond fast to temperature changes and they agree very well to each other. Only very small temperature differences occur (insets of Fig. 3), but they are lower than 1 °C. Such low differences comply with the industrial standard for type S thermocouples [6].

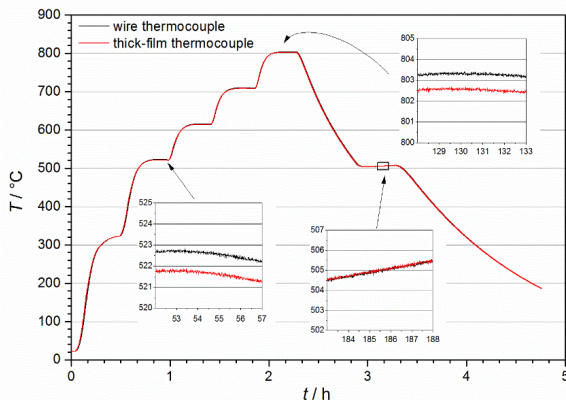


Fig. 3. Thermocouple responses to step-like temperature changes

In the second experiment, the furnace temperatures were changed in cycles between 150 and 800 °C and the responses of both thermocouples were recorded. The test comprised 15 cycles with temperature ramps between 150 °C and 800 °C. Each cycle consisted of a 30 minutes lasting heating period followed by a cooling ramp with a 10 minutes period for stabilization at elevated temperature. The results are given in Fig. 4.

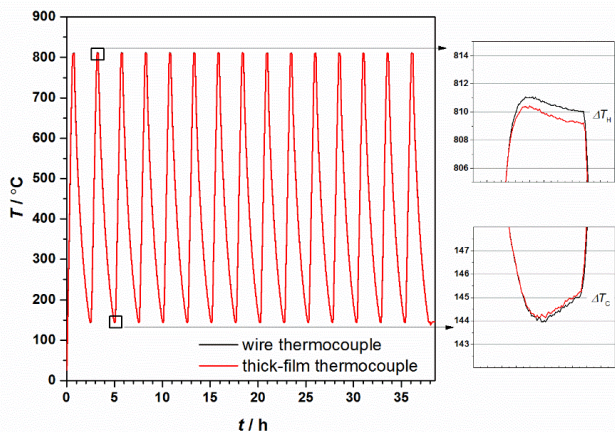


Fig. 4. Cyclic temperature changes. Graphs on the right hand side show exemplarily temperature differences at the highest (ΔT_H) and the lowest (ΔT_C) temperatures.

Both sensors exhibit almost the same and stable characteristics. In the Tab. 1, some statistical values calculated from 15 heating/cooling cycles for both thermocouples are shown. Small differences ($<1\text{ }^{\circ}\text{C}$) between both structures occur only at highest temperature of $800\text{ }^{\circ}\text{C}$, as shown in the small graphs in Fig. 4 and in Tab. 1. Measured temperatures at low level (about $143\text{ }^{\circ}\text{C}$) vary by ca. $\pm 0.13\text{ }^{\circ}\text{C}$ for the wire thermocouples and ca. $\pm 0.15\text{ }^{\circ}\text{C}$ for the thick-film ones, respectively. At high temperatures (about $800\text{ }^{\circ}\text{C}$) the values are a little higher: $\pm 0.56\text{ }^{\circ}\text{C}$ (wire) and $\pm 0.89\text{ }^{\circ}\text{C}$ (thick-film).

Table 1. Statistical values calculated from 15 heating/cooling cycles for both thermocouples. All values in $^{\circ}\text{C}$.

	Mean	Std. deviation	Minimum	Median	Maximum
wire thermocouple low level	143.89	0.0876	143.76	143.91	144.02
thick-film thermocouple low level	143.99	0.0908	143.88	143.97	144.17
wire thermocouple high level	810.92	0.3214	810.32	811.04	811.430
thick-film thermocouple high level	810.50	0.4093	809.31	810.45	811.02

4. Conclusions and outlook

First results of the application of an experimental PtRh composition for thick-film type S thermocouples were shown. The experimental paste was used in combination with platinum ink DuPont 9141R. The screen-printing properties of the new material are similar to standard thick-film pastes. Minimum investigated line width is 150 micrometers; smaller lines may be possible. Both materials were separately fired at $950\text{ }^{\circ}\text{C}$. The obtained structures were heated up to $800\text{ }^{\circ}\text{C}$, and the thermocouple voltages were measured. The output voltages of thick-film thermocouple temperature sensors made of Pt- and Pt/10%Rh-pastes obey the German and European standard DIN EN 60584. Therefore, commercially available readout electronics can be used.

Cyclic stability is excellent. It is assumed that at $600\text{ }^{\circ}\text{C}$, almost no aging occurs. At $800\text{ }^{\circ}\text{C}$, a small running-in behavior can be seen. It is assumed that firing at higher temperatures (pre-aging) and/or a protective cover reduce this effect. Furthermore, it is expected that firing at higher temperatures, will further improve the long term stability at ultra-high temperatures. Since the first tests were very promising, more long-term stability tests will be conducted in the future.

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