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Investigations of the electrical contacting of new piezoresistive polymer-ceramics

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Abstract

Silicon oxycarbide nanocomposite with segregated carbon (C/SiOC) belongs to the group of polymer derived ceramics (PDCs). The C/SiOC shows piezoresistive properties by mechanical load. As an advantage over metal foil strain gauges and doped sensing resistors, the C/SiOC material is expected to have a high gauge-factor (*k*-factor) even for high temperature applications. In order to characterize the piezoresistive properties of the C/SiOC material, force-impedance characteristic must be acequired. The paper will show different layouts of the electrical contacts and different resistance measurement by using four-terminal and two-terminal sensing. The measurement results will be compared to each other and be discussed.

Key words: polymer derived ceramics, silicon oxycarbide nanocomposite, high temperature application, piezoresistive ceramics

Introduction

The Institute of Electromechanical Design (EMK) and the Institute of Material Science (DP) at TU Darmstadt are doing research on new piezoresistive ceramics, which is a part of a DFG project. The new material belongs to the group of polymer derived ceramics (PDCs). The piezoresistive ceramic is made of silicon oxycarbide nanocomposite, which includes segregated carbon (C/SiOC) (Fig. 1) [1]. Preliminary investigations show that C/SiOC has a high temperature resistance performance up to 1400 °C. The C/SiOC also shows piezoresistive properties, which might be caused by segregated carbon that is part of its own components. As an advantage over metal foil strain gauges and doped sensing resistors, the C/SiOC material is expected to have a high gauge-factor (*k*-factor) even for high temperature and aggressive environment [2][3][4].

Carbon-containing silicon oxycarbide nanocomposites C/SiOC

The starting material of C/SiOC is the polymeric precursor (poly (methylsilsesquioxane)) PMS MK, from Wacker AG, München, Germany. It is a preceramic polymer which is cross-linked at 250 °C for 2 h, and then pyrolyzed at 900 °C for 2 h under flowing argon [1]. The new material is ball milled and sieved to a particle size < 100 µm.

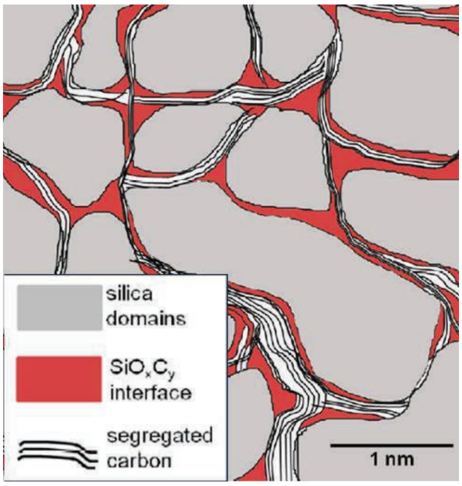

Fig. 1 Illustration of the microstructure of silicon oxycarbide [1]

Subsequently, the sieved powder is hot-pressed at 1600 °C to round and cylindrical pellets (Fig. 2). To obtain dense C/SiOC monoliths, the hot-press process is performed with 30 MPa under argon atmosphere with 30 min dwell time. The Young's modulus of the C/SiOC is about $E = 85$ GPa [5]. The C/SiOC pellets diameter is between 10 and 20 mm and the pellets are 3 mm thick. Finally, the C/SiOC pellets are cut to (3x3x10) mm³ samples for characterization.

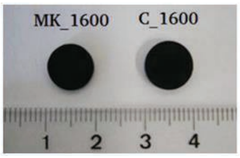

Fig. 2 Samples of the C/SiOC pellets with d = 10 mm diameter.

Measurement on C/SiOC samples

In order to characterize the piezoresistive properties of C/SiOC samples, force-impedance characteristic must be investigated.

The sample is clamped in a special force source made by EMK [6]. The force source has a nominal force range of 40 N. It is driven by a precision stepper motor. A force sensor is integrated to monitor the created force from stepper motor. The force coupling on the stepper motor, the force sensor and the sample is in series. A spring in the force flow acts as a resilience. The measurement uncertainty is under 2 % [6].

The sample is pressed on its front surface *A* with the force *F* (from 0 N to 40 N) which is shown in Fig. 3. Due to the pressure $p = F/A$, the sample gains a negative strain which causes a mechanical stress in the C/SiOC sample. The mechanical stress determines a change in resistance of the C/SiOC sample [2].

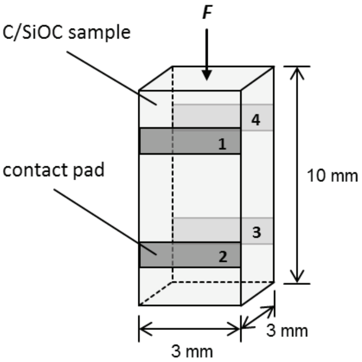

Fig. 3 C/SiOC sample with four contact pads 1,2,3,4.

In order to obtain the change in resistance of the sample, the C/SiOC sample must contact with a measurement system for measuring the change in resistance. The PXIe measurement system from National Instruments measures the signals both from the force sensor and the sample at the same time via a LabVIEW program. The force source is controlled via the same LabVIEW program. Hence, C/SiOC samples must be electrically contacted.

There are different ways to electrically contact the sample. The main point is that the contact pads should be outside of the force coupling to avoid surface effects due to the influence of the force on the sample. We chose two layouts:

1. Four small contact pads (Fig. 3)
2. Two big contact pads (Fig. 4).

The layout with four small contact pads allows a four-terminal sensing of the samples with contact resistance compensation. It is also suitable for two-terminal sensing and measurement of pure longitudinal and transverse effects.

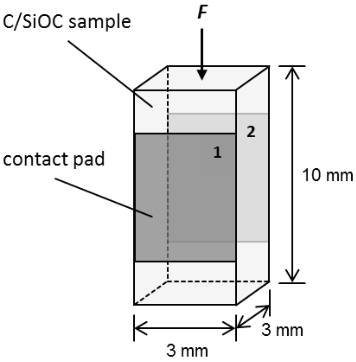

Fig. 4 C/SiOC sample with two big contact pads 1,2.

But due to the small contact area, the signal intensity should be lower than bigger contact area with a homogeneous electrical field. In the other hand, the layout with big contact pads is only suitable for two-terminal sensing and measurement of the transverse effect.

We have investigated three different types of the electrical contacts on the C/SiOC samples.

1. Coating the contact pads by vapor deposition of platinum with a layer thickness of 100 nm. Subsequently, using an isotropic conductive epoxy from type E-Solder 3021 to contact the platinum coated contact pads with tinned wires.
2. Coating the contact pads by vapor deposition of chromium (20 nm) and nickel (150 nm). Subsequently, using an isotropic conductive epoxy from type E-Solder 3021 to contact the coated contact pads with tinned wires.
3. Contacting the sample with tinned wires directly by using an isotropic conductive epoxy from type E-Solder 3021.

All investigations in this paper have been done under laboratory conditions at room temperature. For the measurement, the supply current is constant 1 mA. The maximum load on the C/SiOC samples is 50°N. Measuring cables are ca. 1 m long.

Four-terminal sensing on C/SiOC samples

Four-terminal sensing allows the measuring of the nominal resistance R_p of the C/SiOC sample at room temperature and unloaded force source ($F = 0$ N). For e.g. we connect the contacts 1 and 3 to measure current and connect contacts 2 and 4 to measure voltage. The influences of the contact resistance R_k can be neglected. The investigated C/SiOC samples differ from their segregated carbon in vol%. C/SiOC samples with 17 vol% and 46 vol% segregated carbon have been investigated.

First, we have measured the nominal resistance to determine the influences of the type of the electrical contact. The nominal resistance of the investigated C/SiOC samples with 17 vol% and 46 vol% segregated carbon is shown in the following table:

Table 1 Measured nominal resistance of C/SiOC samples with 17 vol% and 46 vol% segregated carbon without load (four-terminal sensing).

sample	**type of contact pads**	**free C**	**R_p in Ω**
MK_P1	coated with Pt	17 %	3584
MK_P1	directly by epoxy	17 %	3437
MK_P2	coated with Pt	17 %	511
MK_P2	directly by epoxy	17 %	551
MK_P3	coated with Pt	17 %	785
MK_P3	directly by epoxy	17 %	551
RD_688	coated with Cr, Ni	46 %	0.146

The sample RD_688 has the lowest nominal resistance due to its high vol% of segregated carbon. The three samples MK_P1, MK_P2 and MK_P3 were cut from the same pellet. Due to amorphous distribution of the starter powder during the hot press process, it is assumed that the properties of these samples must be the same. But the nominal resistance from the sample MK_P1 differs significantly from the other two samples. The measurement results also show that the type of the electrical contact has insignificant influence of the nominal resistance.

The second investigation shall determine the sensitivity of the piezoresistive effect in the C/SiOC samples. Three measurement results are shown in Fig. 5 for the MK-samples. The force coupling occurs manually. The force-impedance characteristic does not feature clearly, in particular for the samples MK_P1 and MK_P3. The sample MK_P2 shows a piezoresistive effect thus the change in resistance due to mechanical load, but the result is not reproducible. Further investigation of the sample RD_688 shows no piezoresistive effect, either (Fig. 6).

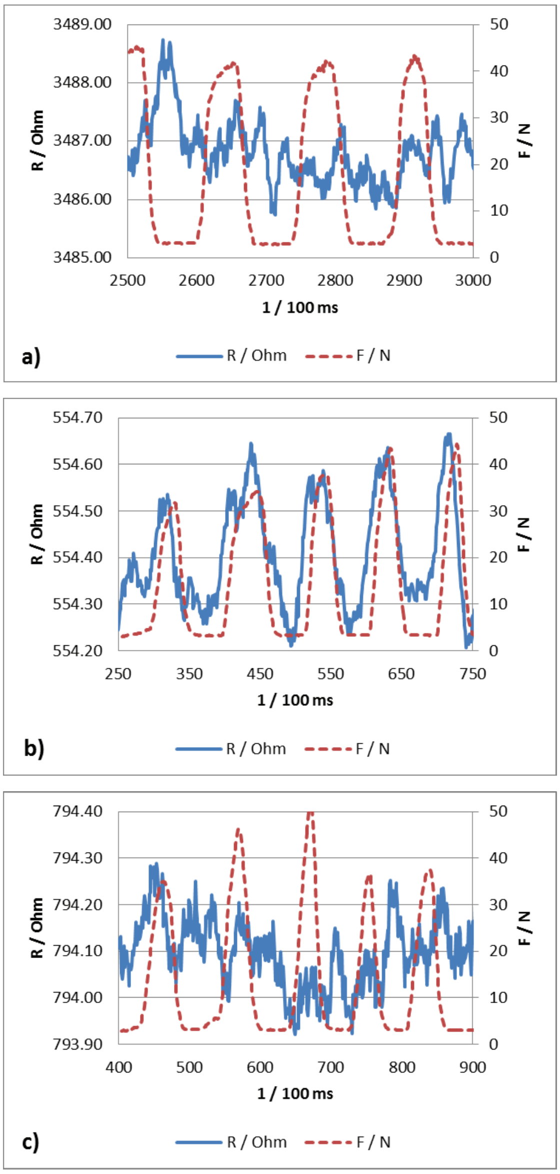

Fig. 5 Diagram of measured force-impedance characteristic of MK-samples with load up to 50 N (extract). a) MK_P1, b) MK_P2, c) MK_P3

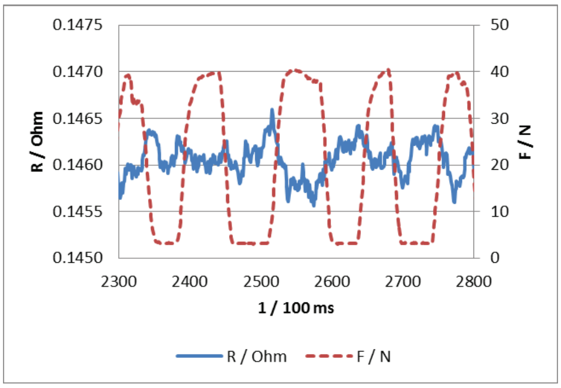

Fig. 6 Diagram of measured force-impedance characteristic of RD_688 sample with load up to 40 N (extract).

Two-terminal sensing on C/SiOC samples

We only use the contact pads 1 and 3 for the two-terminal sensing (Fig. 3). In this case, we obtain a diagonal current path through the C/SiOC sample. Here, the contact resistance must be considered.

Table 2 Measured nominal resistance of C/SiOC samples with 17 vol% and 46 vol% segregated carbon without load (two-terminal sensing).

sample	**type of contact pads**	**free C**	**R_p+R_k in Ω**
MK_P1	coated with Pt	17 %	9858
MK_P1	directly by epoxy	17 %	13774
MK_P2	coated with Pt	17 %	1050
MK_P2	directly by epoxy	17 %	1252
MK_P3	coated with Pt	17 %	2131
MK_P3	directly by epoxy	17 %	2898
RD_688	coated with Cr, Ni	46 %	0.735

Table 2 shows the nominal resistance of the C/SiOC samples with contact resistance. These values are up to four times higher than values of four-terminal sensing. This means that the contact resistance is higher than the nominal resistance of the samples. It also shows that the contact resistance is higher by using the epoxy directly.

The advantage of the two-terminal sensing is that a Wheatstone bridge can be used to gain more stable signals. Fig. 7 shows the change in resistance *ΔR* of the MK_P2 sample due to mechanical load by using a 1/4 bridge. The contact resistors are outside of the force coupling. This reduces the influence of the contact resistance during the measurement. It also ensured that the measured effect comes from the sample and not from the contact resistors.

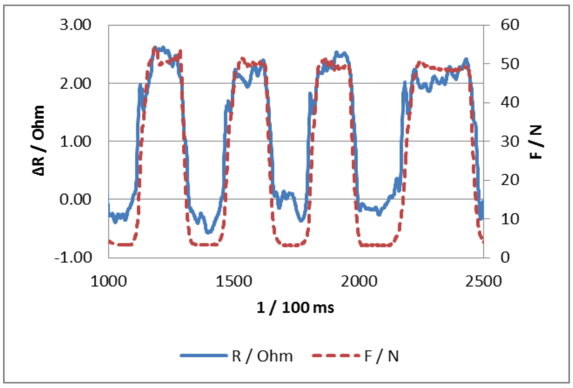

Fig. 7 Diagram of measured force-impedance characteristic of MK_P2 sample with load up to 50 N (extract).

The signal yield has been improved in comparison of Fig. 5 b). Unfortunately this result only applies to MK_P2 sample and it is not reproducible, yet. Other samples do not show any notable piezoresistive effect.

We assume that bigger contact area has a homogeneous electrical field. Therefor we made a new sample RD_212 with 46 vol% segregated carbon with big contact pads. Measurement results of the new sample shows a clearly change in resistance due to mechanical load (Fig. 8). The force coupling was controlled by PC via LabVIEW program.

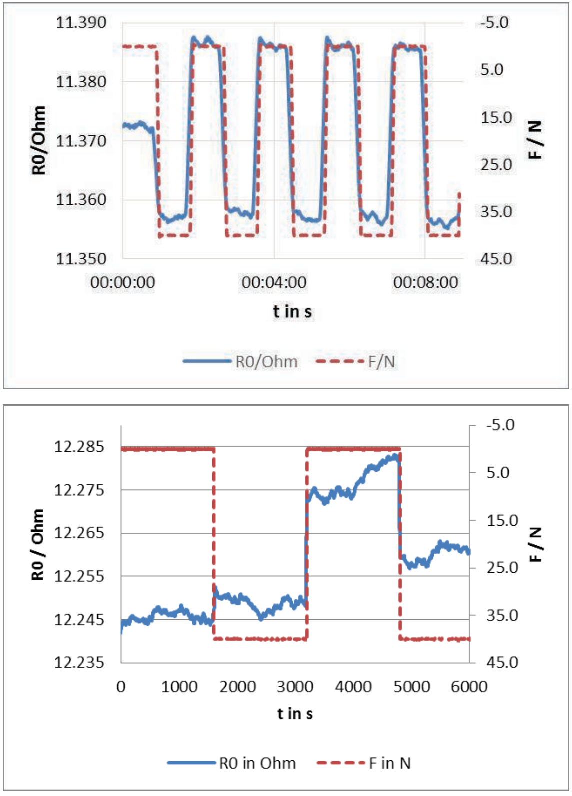

Fig. 8 Diagram of measured force-impedance characteristic of RD_212 sample with load up to 40 N.

This result is not reproducible, either. But the change in resistance due to mechanical load is notable (Fig. 8).

Conclusion

This paper shows different types of electrical contact of the C/SiOC samples. Using the four-terminal sensing, the nominal resistance of the C7SiOC samples can be determined. But due to small contact area, the signal is not stable enough. Electrical contact with bigger contact area has a homogeneous electrical field. This can help to gain more stable signal. But only the transverse effect can be measured. The contact resistance is significant higher than the nominal resistance of the samples. Hence, the contact resistance must be considered for two-terminal sensing. The contact pads should be outside of the force coupling to avoid surface effects due to the influence of the force on the sample.

Further investigations of samples with 17 vol% segregated carbon with big contact pads should be proceed in order to compare the results with small contact pads.

The measurement results are not reproducible, yet. We have two approaches to solve the problem. The first one, the manufacture of the C/SiOC must be more reproducible. Measurement results shows that samples cut from the same pellet do not have the same nominal resistance or the same force-impedance characteristic. Secondly, we must investigate negative effects as surface effects, temperature effect and others to ensure reproducible measurement results.

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