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Polymer composite based microbolometers

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Abstract. This work focuses on the basic suitability assessment of polymeric materials and the corresponding technological methods for the production of infrared (micro-) bolometer arrays. The sensitive layer of the microbolometer arrays in question is composed of an electrically conductive polymer composite. Semiconducting tellurium and vanadium dioxide, as well as metallic silver, are evaluated concerning their suitability as conductive filling agents. The composites with the semi-conducting filling agents display the higher temperature dependence of electrical resistance, while the silver composites exhibit better noise performance. The particle alignment – homogeneous and chain-shaped alike – within the polymer matrix is characterized regarding the composites' electrical properties. For the production of microbolometer arrays, a technology chain is introduced based on established coat-forming and structuring standard technologies from the field of polymer processing, which are suitable for the manufacture of a number of parallel structures. To realize the necessary thermal isolation of the sensitive area, all pixels are realized as self-supporting structures by means of the sacrificial layer method. Exemplarily, 2×2 arrays with the three filling agents were manufactured. The resulting sensor responsivities lie in the range of conventional microbolometers. Currently, the comparatively poor thermal isolation of the pixels and the high noise levels are limiting sensor quality. For the microbolometers produced, the thermal resolution limit referring to the temperature of the object to be detected (NETD) has been measured at 6.7 K in the superior sensitive composite layer filled with silver particles.

1 Introduction

Microbolometers belong to the group of thermal infrared detectors and are used as sensor arrays primarily in thermal imaging devices. In accordance with Planck's law, the measuring of infrared radiation permits passive target analysis as well as non-contact temperature measurements of solid bodies, thus opening a large number of possible applications. In microbolometer detectors, the absorbed infrared radiation causes a change in temperature, triggering a local alteration in resistance within the sensitive area (thermoresistive effect). Conventionally, sensitive resistor elements include vanadium oxide (VO_x), amorphous silicon (a:Si) and ceramic semiconductors (YBCO), which are not usually used in semiconductor production and are difficult to deposit (Ambrosio et al., 2010). Additionally, such microbolometer arrays are manufactured in an elaborate technological process chain, as individual pixels have to be realized as self-supporting structures. This is necessary to ensure a high thermal insulation of the sensitive area and thus great thermal and electrical responsivity. The high cost of microbolometer production is still limiting their widespread use, particularly civilian use, which is highly price-dependent. This motivates the basic research of alternative, economically viable materials and the corresponding technical methods for the production of microbolometer arrays.

The focus of this contribution is on polymer-based materials, as they possess advantages over conventional inorganic materials with regards to both the wider and more cost-efficient material range, and the large number of simple and parallel processing possibilities. A number of publications suggest this approach. Kaufmann et al. describe a possible method for the manufacture of electrically conductive sensitive bolometer layers consisting of ion-implanted polymer

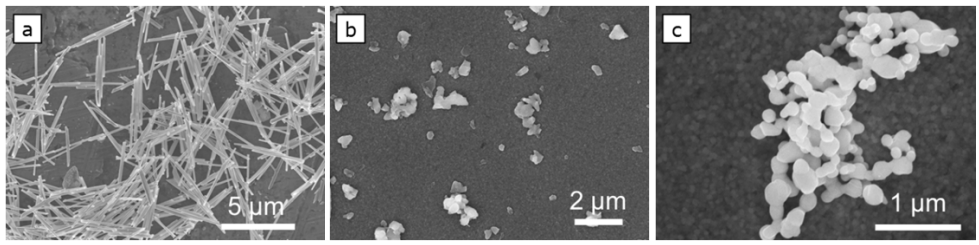

Figure 1. Scanning electron microscope (SEM) views of **(a)** synthesized tellurium needles, **(b)** vanadium dioxide particles, and **(c)** silver particles.

layers (Kaufmann et al., 1996). The electrical properties of the modified polymer layer depend on the ion dose, the ion energy and the ion current density. Liger et al. describe an approach in which the sensitive layer is formed by pyrolyzing the parylene C polymer (Liger, 2006). The pyrolysis of the pre-deposited parylene layer is performed in two stages at temperatures ranging from 660 to 800 °C and causes a share of the benzene rings contained in the parylene C (Liger, 2006) to transform into graphite-like areas. The resistance of the pixel is determined by the rate of graphitization, and thus by the temperature during pyrolysis. Liger (2006) gives a calculated noise equivalent temperature difference (NETD) value for his microbolometer pixels of 31–109 mK. In these works, polymer-based technologies for the manufacture of self-supporting bolometer-pixels are presented. The considerable energy inputs required for the manufacturing methods described limits the application spectrum, particularly with regards to flexible polymeric substrates. Additional works, addressing in particular the production and characterization of polymer-based sensitive bolometer layers, examine the suitability of the intrinsically conductive polymer Poly(3,4-ethylenedioxythiophene)/Poly(styrenesulfonate) (Son et al., 2009) and of carbon nanotubes with (Aliev, 2008) and without (Zeng et al., 2012) surrounding matrix polymer. Best sensor performance for these sensitive materials is given by Zeng et al. (2012) with a calculated detectivity D^* of 1.09×10^7 cm $Hz^{1/2}$ W^{-1}.

In this work, sensitive layers consisting of electrically conductive polymer composites composed of an insulating matrix polymer and a conductive filling material are used. When using such polymer composites, the mechanical, chemical and electrical properties of an individual layer can be adjusted and optimized separately. Especially chemical and thus technological properties are determined by the polymer matrix, as long as the proportion of the filling agent is small enough. The electrical properties are given by the type, structure and distribution of the conductive filling material.

2 Experimental

2.1 Materials

When selecting the materials to be used, the essential technological and electrical requirements of the polymer-based microbolometer arrays have to be taken into account. For the sensitive polymer composites, individual conditions apply for both the polymer matrix and the filling agents. Furthermore, both components have to be chemically compatible in order to form a stable suspension at least for the duration of processing.

2.1.1 Filling materials

Crucial criteria for material choice are the electrical parameters resistivity and temperature coefficient α_R of resistance. Semiconducting materials with a comparatively high α_R of $-(2–5)$ % K^{-1} as well as metallic materials with exceptionally good noise performance show great potential as filling materials. Another requirement results from the individual pixel element's geometry given by the sensitive layer's maximum layer thickness, which should be as small as possible (< 2 µm) to achieve the required low heat capacity of the microbolometer pixel. Therefore, only sufficiently small filling particles, which are also synthesizable in the desired geometric form while still meeting high quality standards, can be used. Considering these criteria, particles composed of tellurium (Te), vanadium dioxide (VO_2) and silver (Ag) have been used within the framework of this research (Fig. 1).

Monocrystalline tellurium is a semiconductor with an anisotropic trigonal crystal structure, giving it a predominant growth direction along the main axis, with the tellurium particles growing as needles. Another effect of its anisotropic structure is an anisotropic behavior of electrical conductivity, which is $\sigma_c = 2$ S cm^{-1} along the main axis at room temperature, while being lower by magnitudes along the other axes (Nussbaum, 1954). Te-needles were synthesized using chemical reduction of telluric acid (H_6TeO_6) with hydrazine (N_2H_4) as presented by Mayers and Xia (2002). The chosen synthesis procedure leads to a clean surface of the particles, which is desired for good electrical contacts. The resulting tellurium needles display a homogeneous size distribution at

Table 1. Electrical properties of the examined materials at room temperature (300 K): band gap E_g; temperature coefficient α_R of resistance and resistivity ρ.

Material	E_g [eV]	α_R [% K^{-1}]	ρ [Ωcm]	Reference
Te (c axis)	0.33	−2.45	2.0	Loferski (1954)
VO_2	0.65	−4.19	≈ 100	Berglund and Guggenheim (1969)
Ag	–	0.41	1.5×10^{-6}	Ashcroft and Mermin (2001)

diameters of 200–250 nm and length of 5–6 µm. The particles' aspect ratio, therefore, is ca. 25.

Polycrystalline vanadium dioxide particles with a size distribution from a few hundred nanometers to ca. 10 µm are commercially acquired with a purity of 99.9 % based on trace metals analysis, according to manufacturer information (manufacturer: Aldrich). By means of a sedimentation process, particles are separated depending on their size. The resulting particles have a maximum size of 2 µm, as can be seen in Fig. 1b), and thus meet the above-mentioned geometrical requirements of filling material.

The silver particles used (manufacturer: Aldrich) were also acquired commercially and have a silver content of at least 99 % and a diameter of ca. 150 nm, according to manufacturer information. The particles have a high defect structure and internal energy, causing a metastable, energetic, activated powder, which may form agglomerates of a size of approximately 1–2 µm.

The electrical properties of these materials are summarized in Table 1.

2.1.2 Polymers

The aspired requirement of realizing the individual sensor structures with just a few simple process steps is most easily attained by using filled photoresists. The structure transfer is performed by UV exposure through a photomask. The respective process step is reproducible and applicable in large scale. Furthermore, many photoresists can be cross-linked, making them chemically stable against solvents and giving them higher mechanical solidity. The cross-linking reaction takes place either directly under UV exposure or by subsequent heat input.

One photoresist with the above-named properties is the AZ 1514 positive photoresist by Clariant, with the polymeric main component being Novolak. This photoresist is used as the polymer matrix of the microbolometer pixel's sensitive layer. The special suitability of the AZ 1514 photoresist stems from its capability to be thermally cross-linked into a phenolic resin in the temperature range 120–160 °C after its lithographic structuring (Roy et al., 2003), which gives it extremely stable mechanical and chemical properties.

Another structuring polymer used is the Pyralin 2722 photoresist by HD MicroSystems. It is a negative photoresist with polyimide (PI) for its polymeric main component. Polyimides are very stable against most solvents and high temperature strains (> 300 °C) (Fukukawa and Ueda, 2008) and therefore suitable for use as permanent, structuring carrier layers, which are created in the first process step of the microbolometer array manufacture.

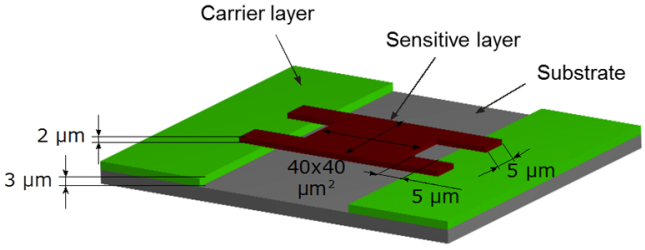

Figure 2. Geometric target parameters for the microbolometer pixel to be realized.

Other polymers used in this work are the heat-cured two-component Sylgard 184 (manufacturer: Dow Corning), with the silicone polydimethylsiloxane (PDMS) being the main component, and a paraffin wax purchased from Aldrich, with a melting range of 70–80 °C.

2.2 Technology

A technology chain was developed for the production of microbolometer arrays whose sensitive layers are made from electrically conductive polymer composites. As the thermal insulation of the sensitive area is key, any pixels have to be realized as self-supporting structures. The construction scheme of a self-supporting pixel is portrayed in Fig. 2.

For the cost-effective production of microbolometer arrays, established coating and structuring standard technologies for polymer processing were used, all of which are suitable for the simultaneous creation of several structures. In particular, the following aspects had to be considered for these polymer technologies:

- It is necessary that the individual polymer layers can be coated homogeneously in the desired thickness range, and easily structured laterally.
- The solvents and developers to be used must not dissolve or macerate the previously produced layers.
- The thermal stability of existent polymer layers has to be ensured at any exposure to heat, which often occurs

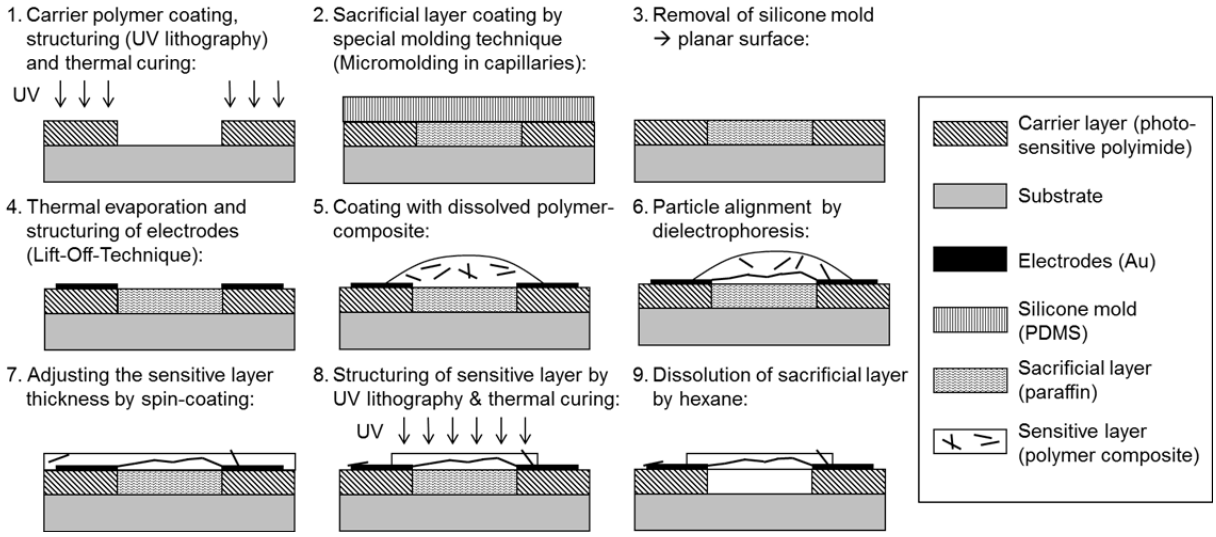

Figure 3. Schematic portrayal of the individual process steps for the manufacture of microbolometer arrays under principal use of polymeric materials and technologies; UV – ultraviolet, PDMS – polydimethylsiloxane.

in the coating of inorganic materials (e.g., the deposition of electrodes).

The derived technology chain consists of seven elementary process steps (Fig. 3). Additional information can be found in Nocke (2011). The technological process is based primarily on the sacrificial layer technology, in which the sacrificial layer is removed during the final process step, creating the self-supporting structure. The micromolding in capillaries (MIMIC) stamping technology was chosen for layer formation and simultaneous structuring of the sacrificial layer (Moonen et al., 2012). The sacrificial layer of heated, liquid paraffin wax fills a pre-structured channel network, owing to capillary action. Due to its low surface tension and viscosity, molten paraffin wax is particularly suitable for this structuring process. Furthermore, it displays favorable solubility properties, as it is only soluble in alkanes and few other solvents. Therefore, it barely limits the choice of possible organic materials in the next process steps.

The electrically conductive networks in the sensitive polymer composites are realized by means of a dielectrophoretic alignment of the filling particles. This effect is based on the particles' polarization in electrical fields and the resulting electrostatic force. In inhomogeneous electrical fields and under suitable conditions, the resultant force causes the desired formation of particle chains between the field-forming electrodes (Pohl et al., 1978; Nocke et al., 2009). The aim of such an alignment is the creation of a conductive network between appropriate electrodes at a preferably low filler loading (here: 0.1 wt. % in relation to the polymer matrix), which is essential for a technological malleability of the polymer composite.

2.3 Characterization

Electrical measurements were performed using the Electrometer 617 with the testbox 8002A (manufacturer: Keithley), which is well suited for high ohmic samples. For temperature-dependent measurements, a climate chamber HC0020 (manufacturer: Voetsch) with additional humidity control was used. All measurements were performed at 0 % relative humidity to minimize humidity influence. Noise and responsivity characteristics were measured with the lock-in amplifier 7265 DSP (manufacturer: EG&G Instruments). In order to establish the sensor parameters of a microbolometer pixel, the frequency-dependent responsivity R_V was measured metrologically by detecting the voltage response of a modulated IR radiation source (manufacturer: DIAS infrared system CS 500 with chopper wheel) with the lock-in amplifier 7265 DSP. The measurement was performed in an evacuated measuring chamber at an ambient pressure lower than 10 Pa.

3 Results and discussion

The main quality parameters of a microbolometer are the (voltage) responsivity R_V, the detectivity D^* and the noise equivalent temperature difference (NETD) (Gerlach and Budzier, 2010). The responsivity of a sensor is defined by the change in its output in proportion to the change of the input parameter and therefore should preferably be high. For a microbolometer, these parameters are the output voltage U_O and the radiation flux Φ_S, respectively. According to the relation

$$R_V = \frac{dU_O}{d\Phi_S} = \frac{\alpha\alpha_R U_B}{G_{th}\sqrt{1+\omega_s^2\tau_{th}^2}} \quad (1)$$

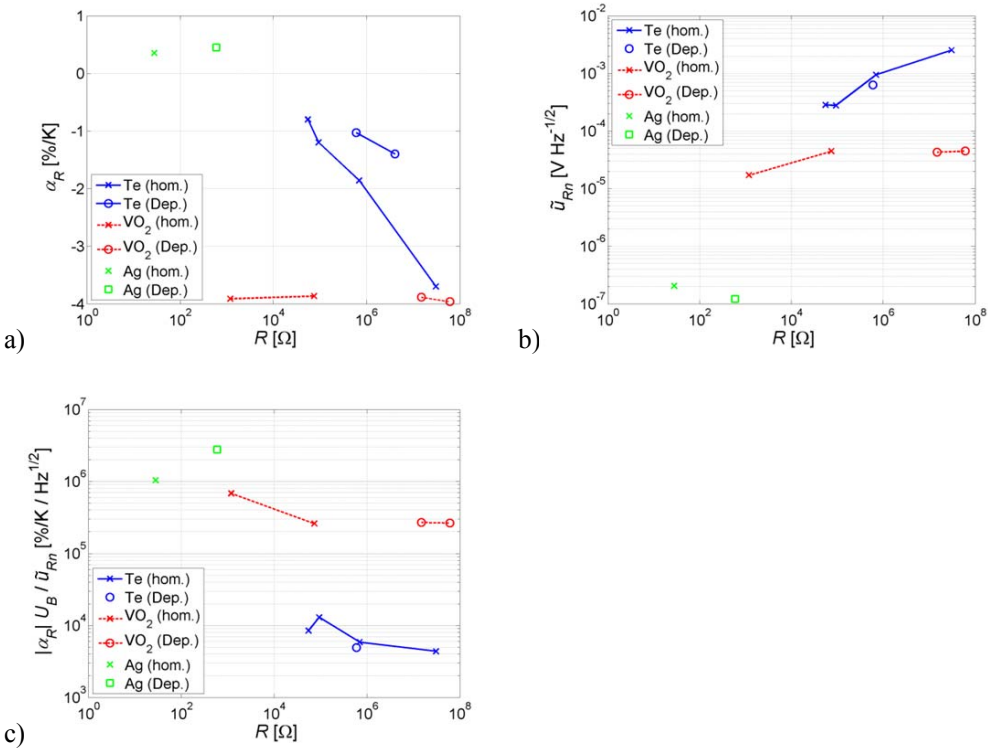

Figure 4. **(a)** Temperature coefficient α_R of resistance and **(b)** spectral noise voltage density $\tilde{u}_{Rn}$ (measured at 52 Hz) and **(c)** influence on detectivity $D^* \propto |\alpha_R| U_B / \tilde{u}_{Rn}$ of a microbolometer, calculated according to Eq. (3) in dependence on the resistance R of the examined polymer composites with homogenous particle distribution (hom.) and particles aligned by dielectrophoresis (Dep.).

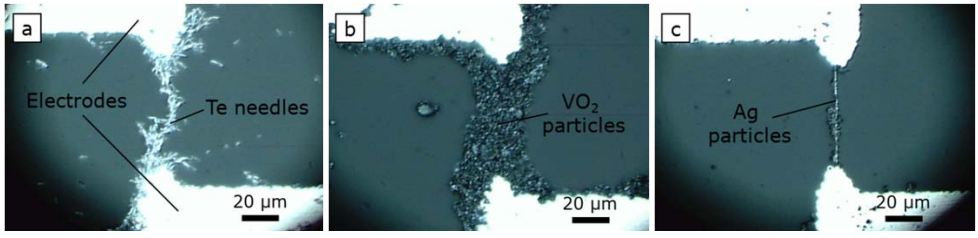

Figure 5. Optical microscopic pictures of dielectrophoretically aligned particles between electrodes: **(a)** tellurium needles, **(b)** vanadium dioxide, and **(c)** silver particles.

with

$$\tau_{th} = C_{th}/G_{th}, \tag{2}$$

the responsivity R_V of a microbolometer depends on the absorption coefficient α, on the temperature coefficient α_R of resistance, on the bias voltage U_B, on the thermal conductance G_{th} between the sensitive area and the surrounding media, on the heat capacity C_{th} of the microbolometer pixel, on the thermal time constant τ_{th}, and on the angular frequency ω_S. The detectivity D^* and the noise equivalent temperature difference (NETD) are sensor parameters typically used to characterize the overall sensor performance by taking into account its ratio of measurement and noise signal. The corresponding basic relation is given by

$$D^* = \frac{\sqrt{A_P} R_V}{\tilde{u}_{Rn}} \propto \frac{1}{\mathrm{NETD}}, \tag{3}$$

where $\tilde{u}_{Rn}$ is the spectral noise voltage density and A_P is the pixel size.

Figure 4 shows the relevant electrical properties of characteristic polymer composites for homogeneously distributed filling particles as well as for composites with particles aligned between the electrodes by dielectrophoresis (Fig. 5).

Stemming from the temperature dependence of the resistance behavior (Fig. 4a) and additional measurements of the current-voltage characteristics (Nocke, 2011), the respective

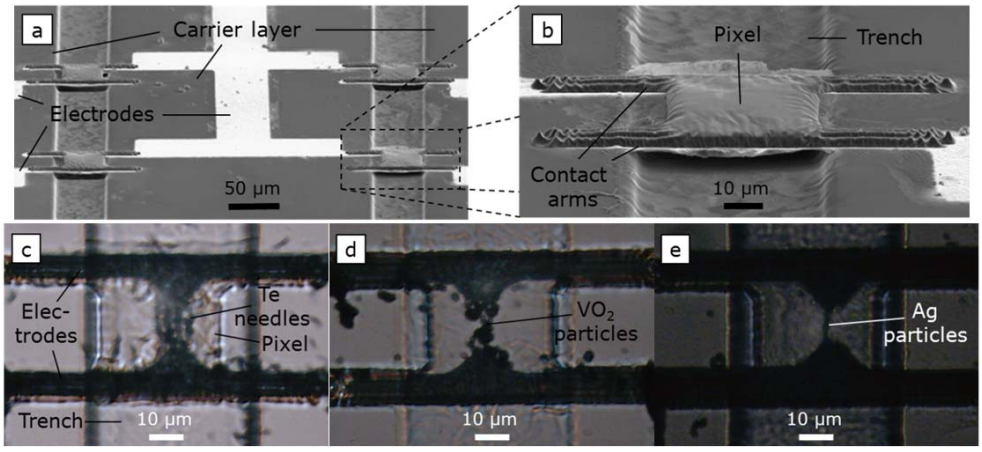

Figure 6. Microscopic pictures of the polymer composite based microbolometers realized: SEM-views of **(a)** the 2×2 array, and **(b)** a magnified individual pixel with a sensitive layer containing aligned silver particles, view of the structures at a tilting angle of 75° across the top view; optical microscopic pictures of individual pixels with aligned **(c)** tellurium needles, **(d)** vanadium dioxide particles and **(e)** silver particles in the sensitive polymer composite.

composites could be connected to individual dominant conduction mechanisms:

– In tellurium composites, the electric conductivity is considerably influenced by potential barriers between the particles. The related hopping conductivity mechanism exhibits a characteristic exponential relation of the resistance R and its temperature coefficient α_R (Mott and Davis, 1979).

– In vanadium oxide composites, the measured temperature coefficient α_R corresponds approximately with the value of the filling material, which is $\alpha_{R,VO_2} = -4.19\,\%\,K^{-1}$. Therefore, the total conductivity of these composites is determined largely by the semiconducting properties of the vanadium dioxide.

– The electrical properties of silver composites are characterized by the metallic conductivity mechanism of the particles, which exhibit a positive and, relative to amount, small temperature coefficient of resistance $\alpha_{R,Ag}$ of $0.41\,\%\,K^{-1}$.

The noise spectra of all composites display significant $1/f$ dependence. The noise levels of silver composites are lower by magnitudes compared to those of composites with semiconducting filling particles (Fig. 4b). The resulting influence on detectivity D^* (Fig. 4c) is greater than the differences in the temperature coefficient of resistance α_R. The detected electrical behavior of the polymer composites has proven largely independent of the variety of particle distribution.

Microscopic pictures of the produced 2×2 microbolometer arrays with the dielectrophoretically aligned filling materials tellurium needles, vanadium oxide and silver particles are shown in Fig. 6. They verify that the technological process is reproducible, independent of the filling material used. The square pixel surface approximately conforms to the specified measurements of $40 \times 40\,\mu m^2$. Figure 6a and b shows tilted scanning electron microscope (SEM) pictures of the microbolometer pixels filled with silver particles, proving the self-supporting nature of these structures. The roughness of the contact arms is caused by diffraction effects occurring during the photolithographic process. The characteristics of the surface topography are examined in the example of a pixel with aligned tellurium needles (Fig. 7). The pixel has the desired layer thickness of 2 µm and runs above the trench without significant bending. The trench has a depth of ca. 4.5 µm.

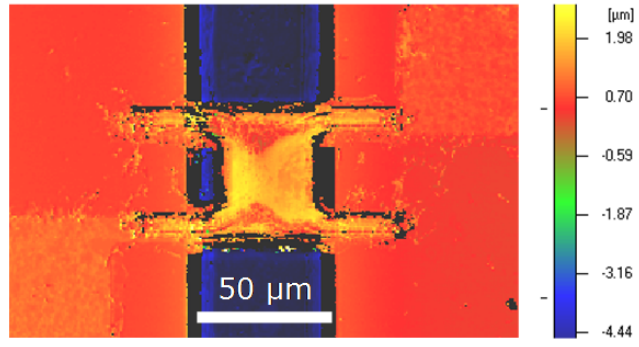

Figure 7. Confocal microscopic view of the surface topography of a microbolometer pixel with a sensitive layer containing aligned tellurium needles.

Table 2 shows the application-relevant electrical and thermal sensor properties (see also Nocke, 2011). The relevant thermal parameters of the microbolometer arrays, thermal conductance G_{th} between the sensitive area, and the surrounding carrier layer and heat capacity C_{th} of the sensitive

Table 2. Electrical and thermal properties of realized polymer composite based microbolometers; calculation of sensor responsivities R_V and detectivities D^* according to the Eqs. (1) and (3); assumed absorption coefficient $\alpha = 1$.

Filling material	Te needles	VO_2 particles	Ag particles
Bolometer resistance R_B (293 K) [Ω]*	$7.5 \pm 1.6 \times 10^6$	$28 \pm 10 \times 10^7$	820 ± 200
Temperature coefficient α_R of resistance (293 K) [% K^{-1}]*	-1.4 ± 0.1	-3.9 ± 0.1	0.51 ± 0.02
Bias voltage U_B [V]*	9	9	0.5
Thermal conductance G_{th} [W K^{-1}]	4.6×10^{-6}	4.6×10^{-6}	4.6×10^{-6}
Heat capacity C_{th} [J K^{-1}]	6.1×10^{-9}	6.1×10^{-9}	6.1×10^{-9}
Thermal time constant τ_{th} [s]	1.3×10^{-3}	1.3×10^{-3}	$1.3 \times 10^{-3}/1.1 \times 10^{-4}$*
Responsivity R_V ($f = 50$ Hz) [V W^{-1}]	2.5×10^4	7.6×10^4	$4.8 \times 10^2/2 \times 10^2$*
Spectral noise voltage density $\tilde{u}_{Rn}$ ($f = 50$ Hz) [V $Hz^{-1/2}$]*	1.7×10^{-3}	8.1×10^{-5}	8.5×10^{-8}
Detectivity D^* ($f = 50$ Hz) [cm $Hz^{1/2}$ W^{-1}]	5.9×10^4	3.5×10^6	2.1×10^7
NETD [K] (Nocke, 2011)	2.4×10^3	41	6.7

* Measured values; statistical values refer to the four pixels of the respective array.

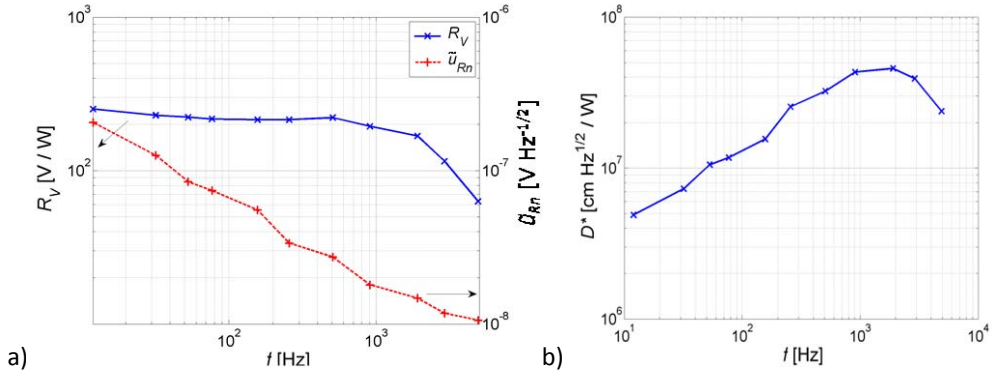

Figure 8. **(a)** Measured responsivity R_V and spectral noise voltage density $\tilde{u}_{Rn}$ and **(b)** calculational derived detectivity D^* for a microbolometer pixel with a sensitive polymer layer containing dielectrophoretically aligned silver particles; bias voltage $U_B = 0.5$ V, bolometer resistance $R_B = 820\,\Omega$.

layer are determined from the pixel dimensions and the respective material parameters. The calculated thermal parameters results in a thermal time constant of $\tau_{th} = 1.3$ ms. The responsivities R_V of the produced microbolometer arrays are calculated according to Eq. (1), using the electrical parameters of bolometer resistance R_B, temperature coefficient of resistance α_R, and bias voltage U_B determined in practice, as well as calculated thermal parameters. The responsivity values of the composites filled with tellurium and vanadium oxide filling materials are approximately two orders of magnitude above those of sensitive silver composites. This difference results from the smaller (according to amount) temperature coefficient of resistance α_R and the lower bias voltage U_B for the values with silver composite.

For the given similar geometric structures, the relation of the individual distinctive sensor parameters results from the electrical properties of the respective polymer composites shown in Fig. 4. Thus, the silver composites, due to their little noise behavior, have highest detectivity D^* and therefore the smallest NETD. The microbolometer arrays with vanadium dioxide composites display the greatest responsivity R_V due to their high temperature coefficient of resistance α_R.

The sensor characteristics of a microbolometer pixel in terms of responsivity and spectral noise voltage density were measured exemplarily on a pixel structure filled with silver particles (Fig. 8a). At low frequencies, the detected responsivity is approximately frequency-independent with a value of ca. $R_V = 2 \times 10^2$ V W^{-1}. The distinct low-pass behavior sets in at a critical frequency of ca. 1.5 kHz, corresponding to a (thermal) time constant of $\tau_{th} = 0.11$ ms. The subsequent detectivity of the measured pixel was calculated according to Eq. (3). As can be seen in Fig. 8b, it shows a strong frequency-dependence affected by the $1/f$ noise characteristic and the dynamic behavior of the responsivity.

While the measured responsivity is in a range comparable to the previously calculated one, the measured time constant is approximately one order of magnitude smaller than the calculated one (Table 2). The deviations can be traced back to erroneous estimates of thermal influences or the presumption

of an optimum wavelength-independent absorption coefficient.

In comparison to values from the relevant literature, which places conventional microbolometers' NETD values at around 30–100 mK (Gerlach and Budzier, 2010), fundamentally higher NETD values were observed. One essential reason for this lies in the higher thermal conductances of the contact arms of the design presented here. Furthermore, the noise levels of polymer composites with semiconducting filling particles are higher than those of semiconducting sensitive layers in conventional microbolometers. The same relation holds true with regards to the organic sensitive layer consisting of pyrolyzed parylene C (Liger, 2006). The NETD value comparison of microbolometers with metallic sensitive layers shows the smallest differences: NETD = 500 mK (microbolometer with sensitive titanium layer: Mansi et al., 2003) as opposed to NETD = 6.7 K (microbolometer pixel with silver composite). These metallic sensitive layers are distinguished by their very low noise levels.

4 Conclusions

The aim of this work was the basic suitability assessment of polymeric materials and related technological methods for the production of polymeric materials for the manufacture of microbolometer arrays with a sensitive layer of electrically conductive polymer composites. The all-polymer compatible technology chain is an innovative approach to the manufacture of polymer-based, self-supporting MEMS (microelectromechanical systems) structures and allows for a prospective economization potential as well as highly parallel processing suitability. Concerning their suitability for use as sensitive layers in a microbolometer pixel, additional metrological and physical observations were made regarding the electrical properties of polymer composites filled with either tellurium needles, vanadium dioxide particles or silver particles.

The best noise equivalent temperature difference NETD, which is similar to the temperature-dependent resolution limit of the measuring object, was detected for the microbolometer array with a sensitive silver composite layer, with its peak value at 6.7 K. This opens new applications for low-cost thermal imaging devices targeted at simple object detection.

In the future, the relevant sensor parameters have to be further improved in order to ensure a proliferation of possible applications. This may be attained particularly well by an enhancement of thermal insulation of the individual pixels and a reduction of noise levels in the composites with semiconducting filling particles. For a better thermal insulation of the individual pixels, the geometric properties of the contact arms have to be adapted to lower their thermal conductance. This requires longer contact arms with a smaller cross section, which mechanically destabilizes the self-supporting pixel structures. Thus, future inquiries will have to aim at further optimization of the polymer-based manufacturing process for microbolometer arrays described herein. Concerning the reduction of noise levels in the composites with semiconducting filling particles, the number and energetic height of potential barriers within the conductive network have to be reduced, as they are major factors in the noise behavior between particles. One possible approach to this is to perform the chemical synthesis (of the sensitive semiconductors) in the vicinity of the microbolometer pixels themselves, leading to barrier-free conducting paths with reduced bolometer resistances. An alternative is offered by depositing the thermoresistive layer in a separate step below or above the self-supporting polymer layer. With such a constructional approach, the simple technology chain developed in this project could be used for the manufacture of self-supporting polymer layers. Thanks to the relevant photoresist's great temperature stability for organic materials, conventional deposition methods known from CMOS technology could be used, as applicable.

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