

(Revised + extended version of paper #8540 presented at Eurosensors XXIII conference; revision 1)

Formulation of Composite Resistive Pastes for Fabrication of Micro-Heaters

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Abstract

We investigate in this work the formulation of composite resistive pastes based on epoxy resins and graphite for micro-heater manufacturing via thick-film technology. These resistive pastes are designed for screen-printing onto a printed circuit board (PCB) substrate; further coating with expandable polydimethylsiloxane (PDMS), a composite based on an elastomeric matrix and expandable microspheres, results in one-shot thermal actuators allowing pumping and sealing in disposable microfluidic lab-on-a-chip devices. The resistive paste must therefore have controlled rheology and resistivity, and exhibit good temperature stability in order to allow high heating power densities. This paper details the formulation and characterisation of suitable epoxy-graphite resistive composites, and the control of their properties through solvents.

Keywords: epoxy resins; graphite; PDMS; thick-film; microfluidics

1. Introduction

Micro-fluidic devices are often based on clean-room processes and materials, e.g. thin-film deposition onto silicon or Pyrex® glass, which are unsuitable for very high-volume disposable devices. Recently, a move towards polymers is observed, as their properties such as transparency, low-cost and biocompatibility make them particularly interesting for micro-fluidic applications. Deposition of devices onto polymer is best done by printing processes such as screen printing and inkjet, depending on the desired materials. One type of envisioned device consists of one-shot valves and pumps to extract and analyze small blood samples and

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using heated PDMS / expandable microsphere composites for actuation [1,2]. However, directly using the copper conductive tracks as heaters [3] has the disadvantage of too low resistance values and low lateral resolution due to the copper tracks acting as heat spreaders. Therefore, we have endeavoured in this work to use polymer / graphite resistive pastes combined with thick-film technology. Graphite was chosen over carbon black as filler as it presents several advantages such as a much higher electrical and thermal conductivity, which allows, in principle, corresponding properties in its composites. However, due to its strong anisotropy, the physics of these composites are complex and not fully understood to date. Carbon-polymer resistors with both thermoplastic [4-7] or thermoset [8-14] matrices, have been investigated previously and can be adjustable in resistivity depending on the filler concentration and type (various grades or mixes of carbon black, graphite or carbon fibre). These systems are known as percolative materials; their conductivity is often observed to follow, slightly above the percolation threshold, a power law of the form [15,16]:

$$\sigma \approx \sigma_0 (x - x_c)^t \quad \text{for } x > x_c \quad (1)$$

where σ represents the bulk conductivity of the composite, σ_0 a proportionality constant, x the volume concentration of the conductive filler phase and x_c the critical value of x for percolation to occur, i.e. the percolation threshold. The percolation threshold x_c is the value of x where conductivity due to the particle network overtakes that of the matrix [17]. Both x_c and σ_0 depend on the microscopic details (microstructure and mean interparticle junction conductance), whereas the critical exponent t is supposed to be independent of the material ($t = t_0 \approx 2$ in three-dimensional lattices). However, the universality of t is limited; ca. half of reported experimental results exhibit $t \neq t_0$ [18], which can be explained by mechanisms such as the tunnelling-percolation model [19], where interparticle conduction occurs by tunnelling, which is likely in our case.

In spite of their advantages, polymer/graphite materials present several issues we have to address. First, standard graphite tends to have a high percolation threshold in polymers, resulting in a high viscosity that can be problematic for both screen-printing and bulk polymer compounding processes. Several types of graphite were tested for conductive fillers, as well as different solvents in order to obtain an adapted formulation of the composite. Temperature stability of the fabricated resistors is also an important issue, and is limited by the glass transition temperature (T_g) of the epoxy matrix. T_g lies at ca. 100°C for “standard” epoxies, i.e. in our range of application. Therefore, composites were formulated in this study using both “standard” and high- T_g epoxies for comparison.

The overall purpose of this study is therefore to obtain the most adapted formulation for polymer/graphite resistive pastes, having a rheology compatible with screen printing, a low resistivity and temperature stability at 100°C or above. As this work mainly concentrates on the formulation of the resin, we chose a graphite filler with a low degree of shape anisotropy.

2. Experimental

2.1. Graphite and epoxy resins

Formulation of thermally stable polymer/graphite resistors is here the “key factor” for a suitable application in a microfluidic device as it is directly connected to the T_g of the matrix. Different materials were tested to give a large array of potential composites design. Three nominal graphite sizes of the same TIMREX® series (provided by TIMCAL, Switzerland) were used for composite manufacturing in order to see the impact on resistivity and rheology. These materials are all “conventional” graphite and look like a fine black powder constituted of ca. ellipsoidal particles with a density of 2.3 g/cm³. Table 1 gives their main properties. The nominal particle size means that 95% of the particles have a major axis smaller than 4 μm (resp. 15 μm and 44 μm). Two epoxies, whose properties are given in Table 2, were used for the tests: EpoTEK® 377 and Martens Plus® provided resp. by Polyscience AG and Swiss Composites (Switzerland).

Table 1. Properties of graphite powders (supplier data)

Name	Particle size (μm)	Specific surface area (m ² /g)
KS4	4	26
KS15	15	12
KS44	44	9

Table 2. Main properties of epoxy resins (supplier data).

Name	T _g (°C)	Viscosity (mPa·s) at R.T.	Curing schedule
EpoTEK® 377	~ 95	150-300	2h at 150°C
Martens Plus®	~ 200	2300	24h at 100°C + 15h at 230°C

2.2. Basic epoxy-graphite composites

A first series of straight resin-graphite composite inks, using the three types of graphite with an EpoTEK® 377 matrix, was prepared by mixing with a three-roll mill (same procedure used throughout this work), in order to determine the effect of particle size on resistivity as well as the volume fraction needed for our application. Test resistors were screen-printed on alumina substrate metallised with thick-film Au terminations. After curing, resistivity was measured through a four-point probe. Cured thickness was typ. 50 μm.

2.3. Formulation with solvents

Depending on the application, high conductivity can be desired, resulting in higher filler loadings. However, because of processability reasons, these higher loadings cannot always be achieved with simple resin-solid systems, requiring the addition of solvents in order for the inks to remain processable; solvents have a double effect on the composite: the dilution decreases the viscosity of the matrix, and the filler concentration in the "raw" composite decreases. Ideally solvents should be miscible with the resin, but remain inert, without chemical reactions. Miscibility can be easily predicted from Hansen solubility parameter theory [20,21]. Concerning reactivity, epoxies are known to have reactivity towards –OH groups, which leads us to avoid alcohols as solvents. The last important point is the boiling point, which should be high enough to avoid the formation of bubbles within the coating during the heating phase. Moreover, for screen-printing, high boiling points (in the 200°C range) are preferred, as they avoid premature drying of the ink. However, the boiling point should also be low enough to fully evaporate the solvent during the resin curing phase. Considering all these requirements, a range of esters and ethers were therefore selected and tested as potential solvents. Table 3 sums up their relevant characteristics and Figure 1 presents their vapour pressure vs. temperature. In the experiments, A/B0 will refer to the reference sample, i.e. without any solvent. Triacetin was not tested in this study, and is given only for comparison purposes as a high-boiling ester.

Table 3. Properties of tested solvents [22]. T_b = boiling point; $P_{150^\circ\text{C}}$ = vapour pressure at 150°C; M = molar mass; d = density.

Name	Type	CAS-n°	T_b (°C)	$P_{150^\circ\text{C}}$ (kPa)	M (g/mol)	d (kg/m ³)	Symbol
Diglyme	Ether	111-96-6	162	76	134.17	943	A/B1
Triglyme	Ether	112-49-2	216	11	178.23	986	A/B2
Tetraglyme	Ether	143-24-8	275	1.8	222.28	1'009	A/B3
Dibutyl carbitol	Ether	112-73-2	256	4.2	218.33	885	A/B4
Dipropylene glycol dimethyl ether	Ether	111109-77-4	175	49	162.23	903	A/B5
Amyl acetate	Ester	628-63-7	149	103	130.18	876	A/B6
Propylene glycol diacetate	Ester	623-84-7	191	30	160.17	1'050	A/B7
Triacetin	Ester	102-76-1	258	2.3	218.20	1'160	/

In preliminary studies, we already had successfully tested ethylene glycol dimethyl ethers, also known as glymes, as solvents for epoxies. These compounds find wide uses in the industry, as inert solvents for chemical synthesis and coatings. They gave promising results, but di- and triglyme are known to be toxic [23-25], and the water affinity of these glymes is rather a drawback, as they may pick up undesired moisture from the air during processing. Therefore it is of great interest to find less problematic substitutes to these compounds, especially for di- and triglyme. The abovementioned solvents were tested through measurement of the weight loss during curing of the resin and the resistivity of the resulting composites.

Composites in this series were made using the same “standard” epoxy as in 3.1, EpoTEK® 377, loaded with graphite KS4 at 10% and 20% volume fraction. Then, ca. 20% (mass) of solvent was added and mixed to the paste.

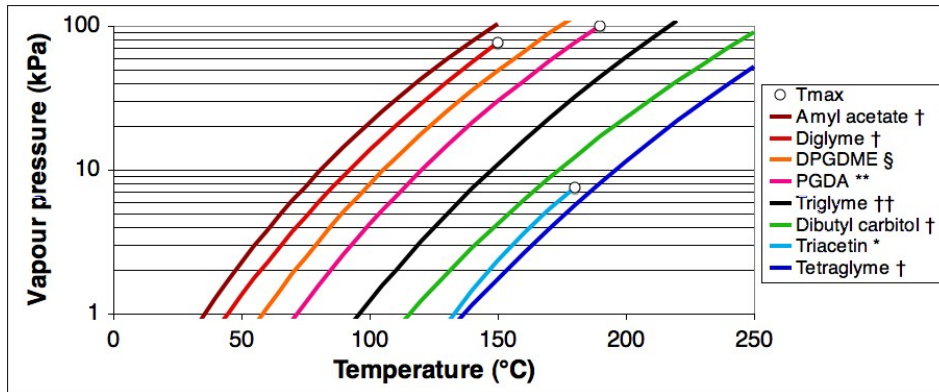


Fig. 1. Vapour pressure vs. temperature (calculated by the Antoine equation [26,27]). † [27]; * [26]; § dipropylene glycol dimethyl ether, approximate data from [28]; ** propylene glycol diacetate, data from [29] refitted together with boiling point; †† interpolated between di- and tetraglyme and adjusted to known pressure at 162°C [30]. Tmax is the maximum validity temperature indicated for the fits in the references.

2.4. Thermal stability and TCR

Finally, a last experiment was carried out to assess the thermal stability of our composite, with the aim to achieve a low drift of the resistivity up to 140°C. For this test, two compositions were prepared, one with the standard EpoTek® 377 resin (c) and the other with a special Martens Plus® resin formulated for high-temperature stability (d). In both cases, KS4 graphite was added to give 20% volume in the final resin and ca. 10% mass diglyme solvent was added to lower resin viscosity.

Stability was qualified by performing three full thermal cycles between 30 and 140°C on the test resistors, with resistivity measured each 10°C. Also, the temperature coefficient of resistance (TCR) was measured between 30 and 65°C, i.e. safely below the lowest T_g .

3. Results

3.1. Basic epoxy-graphite (solvent-less) composites

The room-temperature resistivities of the first series of composites (EpoTEK® 377 matrix with all three graphite powders) are given in figure 2.

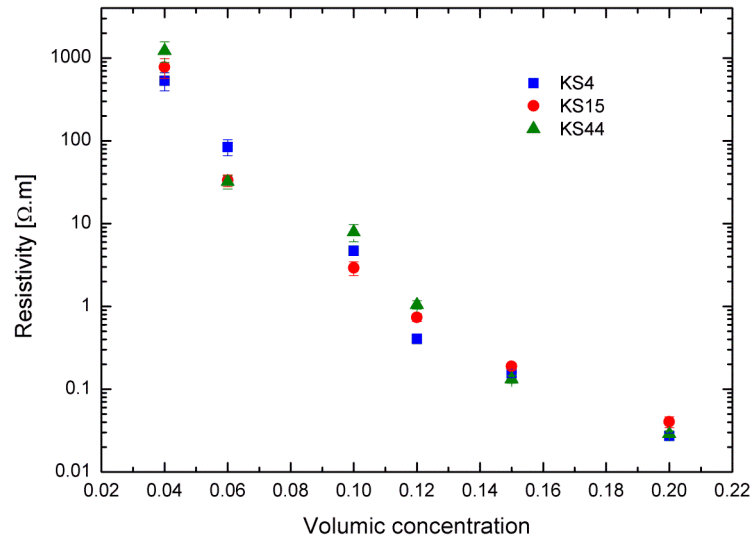


Fig 2. Resistivity of the composites vs. graphite filler volume fraction in EpoTEK® 377, for the three graphite powder sizes.

Several conclusions can be drawn from this graph. First, particle size clearly appears to have no significant effect on resistivity, the tendency of the curve as well as the values remaining the same for the three types, in general agreement with studies with fillers with low to moderate geometric anisotropy [5]. Therefore, we decided to use KS4, the finest powder, for the other tests, as this maximises reproducibility (i.e. the film gets closer to an “infinitely large” system) and allows printing with higher resolution (compatibility with very fine screen meshes). Secondly, it was specified that the application required a low resistivity, e.g. a high filler volume fraction. Based on this first set of values, we decided that a 20% volume concentration in conductive filler was an adequate compromise between low resistivity and processability. Finally, we note the critical volume fraction for percolation is quite low $< 4\%$, but wide critical fractions are found in the literature, even for graphite of low geometric anisotropy [4,5,7,10,12,13], which can be due to various processing factors such as aggregation, or even small debris [17].

3.2. Formulation of the inks with solvents

The room-temperature resistivity of the composites formulated with solvents is given in figure 3, as a percentage of the "solvent-less" sample. Solvents strongly decrease resistivity, which is a desired effect at high filler loadings, where maximal conductivity is desired. We suppose the increased fluidity allowed by the solvent facilitates contact between the graphite particles, an effect also driven by the contraction of the matrix brought about by the evaporation of the solvent. This effect is very marked at higher filler loadings, as shown in figure 2. In this case, there are much more graphite particles, thus, with appropriate fluidity of the paste, it becomes easier to create conductive paths between particles, resulting in a higher conductivity of the final composite. It must also be noted that working with the reference (without solvent) sample becomes difficult at higher loading, as for instance at 20% volume fraction in graphite; processing of even higher loadings would only be possible through solvent addition.

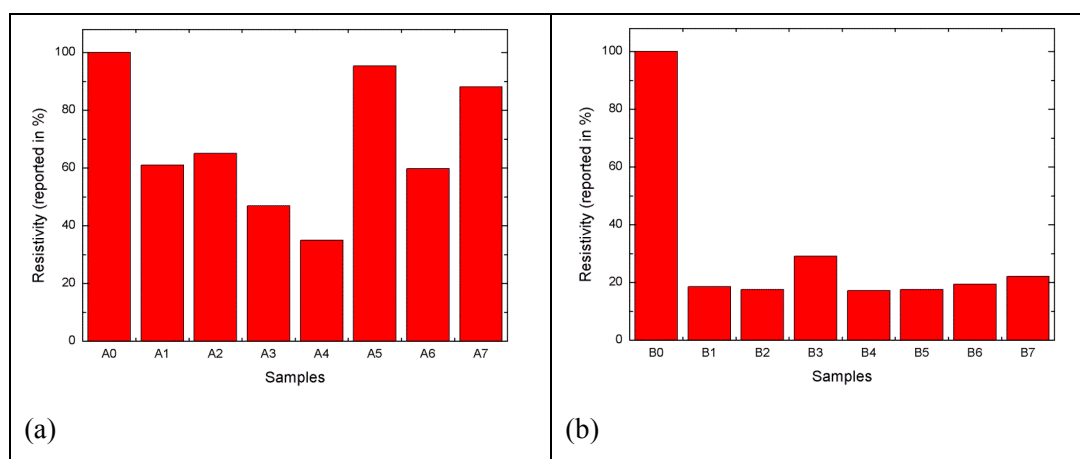


Fig 3. Resistivity of composites loaded with graphite at 10% (a) and 20% (b) volume fraction.

Moreover, adequate solvents for epoxies must evaporate completely during the curing process of the composite (2 hours at 150°C in the oven), which was checked through mass loss measurements. The composite mass was measured at 30, 60 and 120 minutes, and the results are reported in figure 4. Several remarks can be drawn from the graphs. First, the reference sample shows a loss, probably corresponding to water evolution. As far as the A series (10% volume fraction in graphite) is concerned, we can notice that in each case, at least 80% of the total mass loss occurs in the first 30 minutes, not to say almost all of the loss for samples 1, 5 and 6. This result is ascribed to the high vapour pressure of the corresponding solvents (see Figure 1): Solvents 2 & 7, and especially 3 & 4, have low vapour pressures, and thus slower evaporation. This trend is also observed for composites loaded at 20%, where the evaporation process, however, is slowed down by the presence of a higher fraction of filler particles, which increases the tortuosity of the diffusion paths to the surface.

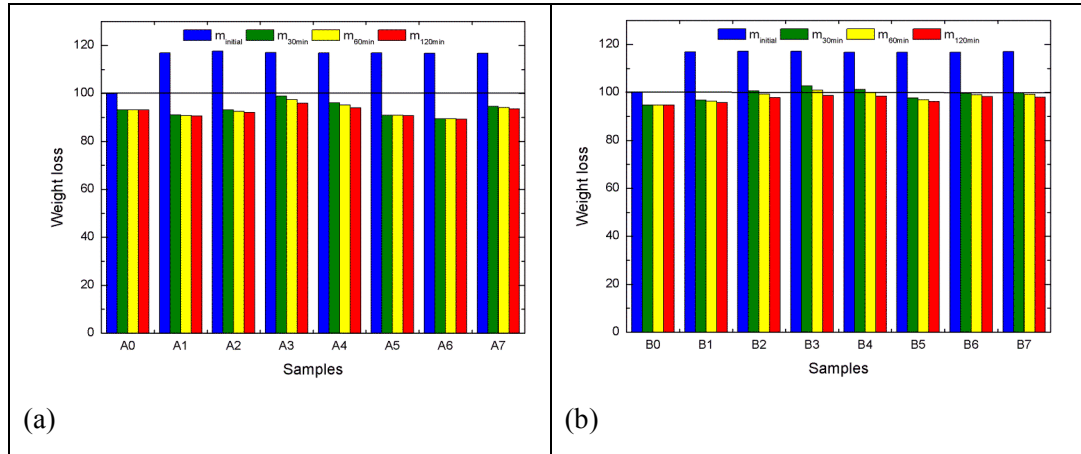


Fig 4. Profile loss mass for samples loaded in graphite at 10% (a) and 20% (b) volume fraction.

3.3. Thermal stability and TCR

Figure 5 gives the result of the thermal stability tests. For EpoTEK® 377 (Fig. 5c), we can see appreciable amounts of hysteresis in the curves, with the first cycle exhibiting a large evolution. A transition is clearly observed at 60-80°C, roughly corresponding to the T_g of the matrix. Above T_g , several effects are in competition: thermal expansion, mobility (charge carriers, bonds between crosslinks) and lower elastic modulus with higher Poisson coefficient. The resistivity of the composite is therefore unstable in the range of our application, compelling us to choose epoxy Martens Plus® (Fig. 5d), which has a higher T_g , to shift this undesired effect to higher temperatures. Thermal cycling between 30 and 140°C of test resistors demonstrated that this final composite is more stable with temperature, although significant change is still observed in the 1st cycle. Stability could be further improved by substituting part of the graphite with carbon black [12-14]. The presence of both fillers also in principle allows a lower resistivity at high loadings [7,13].

The TCR values, measured in the 30-65°C range where both resins are stable, are given in Table 4. A higher TCR magnitude (positive or negative) is desirable to achieve temperature control through the resistor value, and will be sought in future work by adjusting the formulation. It must be noted that TCR is strongly influenced by the substrate thermal expansion through the piezoresistive effect [31]; final TCR values on PCB are likely to be different.

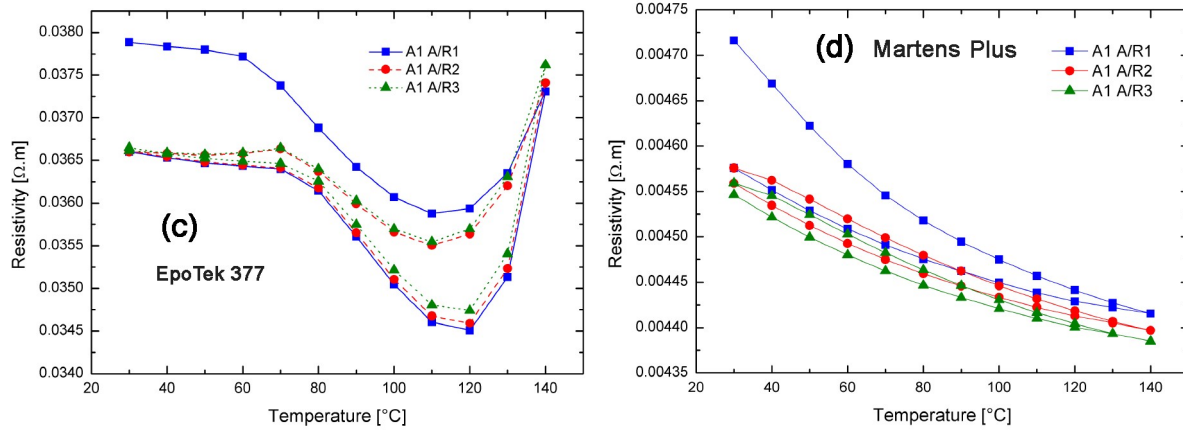


Fig 5. Traces of three thermal cycles for 20% KS4 in (c) EpoTEK® 377 and (d) Martens Plus®.

Table 4. TCR (30-65°C) of the samples on alumina substrates.

Sample	TCR average \pm std. dev. (ppm/K)
(c) EpoTek® 377 + 20% KS4	-150 \pm 13
(d) Martens Plus® + 20% KS4	-320 \pm 5

3.4. Application

The use of a high- T_g resin, a high graphite loading and solvent to allows processability – formulation (d) in previous section – allows stable, low-value resistors apt to be used as micro-heaters for overlying expandable PDMS films (Fig. 6). In a separate study [32], it was determined that such heaters need to reach ca. 110°C to safely heat the overlying PDMS above its expansion threshold of 80°C, and that the required power on a typical PCB was ca. 500 mW for 1 mm square resistors. Formulation (d) with 50 μ m cured thickness yields roughly 100 Ω , giving very reasonable supply parameters of ca. 70 mA at 7 V. In the light of the results of section 3.2, diglyme may of course be substituted by a less toxic compound.

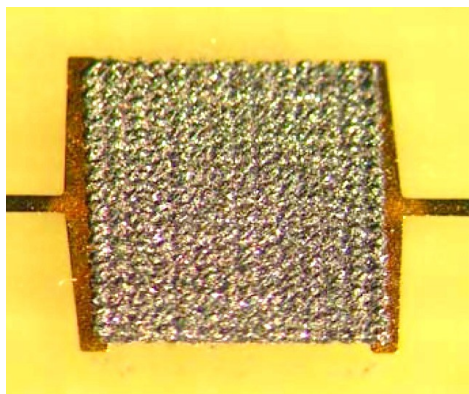


Fig 6. Heating resistor on PCB, 3 mm square [32].

4. Conclusion

In this work, the resistivity and stability of epoxy-graphite composites were investigated. We achieved control of the paste rheology by tuning the formulation with appropriate solvents, which remain inert with the matrix and evaporate in a controllable fashion during the curing. Different esters and ethers were tested, giving a large array of potential solvents with evaporation properties adaptable to the epoxy curing conditions. We also found substitutes to the toxic glymes that were previously used in our application. The control of the paste rheology allows high filler loadings, necessary to obtain low resistivity values. It was also demonstrated that particle size has almost no effect on resistivity, which is in accordance with theoretical models. We thus decided to use graphite with the smallest particle size to maximise printing resolution and reproducibility. The T_g of the matrix was shown to have a pronounced effect on the temperature stability. Thermally stable heaters were therefore successfully formulated using an epoxy matrix with a high T_g , yielding resistors well suited for the manufacture of micro-heaters by economical printing processes. With further improvements in formulation and provided the achievable resolution (ca. 100 μm) is adequate for the envisioned application, this technology constitutes a promising alternative to costly clean-room processes.

Acknowledgements

The authors are indebted to M. Garcin for the fabrication of the samples and to TIMCAL for the graphite powders. This work was partly financed by the Swiss CTI innovation promotion agency.

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5. Biographies

Nathalie Serra graduated in September 2007 (master degree in chemistry) at the “École Nationale Supérieure de Chimie de Lille” which is part of the French “Grandes Ecoles” network, and is pursuing a PhD in resistive nanocomposites at the EPFL.

Thomas Maeder pursued with a PhD in piezoelectric thin films, and a post-doc at IBM Rüschlikon in single-crystal conductive oxides, after graduating from the Ecole Polytechnique Fédérale de Lausanne (EPFL) in materials science. He now heads the thick-film technology group at the EPFL, where current areas of interest are thick-film and LTCC technology for advanced sensor and packaging applications, and sensor networking.

Pierre Lemaire received a combined Master degree in Microtechnology from the Ecole Polytechnique Fédérale de Lausanne (EPFL), the Politecnico di Torino, and INPG of Grenoble, and studies the application of PDMS devices for microfluidics within Debiotech.

Peter Ryser received a Master degree in Physics (University Neuchâtel 1979), a PhD in applied Physics (University Geneva 1985) and a Masters Degree in Corporate Management (Lucerne 1993). His professional background includes several R&D activities. From 1990-1998 he was the head of research at Siemens Building Technologies. Since 1999 Peter Ryser is Professor at the Swiss Federal Institute of Technology EPFL in Lausanne and act as a director for the micro engineering section.

6. Figure captions

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7. Table captions

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