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Carbon based conductive photoresist

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Abstract A conductive photoresist for photolithographic application was studied here. The negative near-UV sensitive epoxy-based photoresist was used as a polymer matrix and conductive carbon black was used as functional filler. DC electrical resistivity of composite as a function of filler concentration has a well-known S-shape. After UV-exposure the resistivity of the composite decreases for almost five orders of magnitude, mostly at percolation threshold (approx. 0.6 vol.%). This effect can be attributed to the fully cross-linked polymer structure formed during UV-exposure of the composite. The resistivity of prepared samples also depend on the state of dispersion of the functional filler obtained using different dispersing additives. Composites with better dispersed particles have lower resistivities. This effect remained below one order of magnitude and decreased after UV-exposure. The composites with carbon black concentration of up to 1.1 vol.% are suitable for spin-coating and photolithography.

Introduction

Electrically conductive polymer composites are used for electromagnetic interference shielding, electrostatic discharge protection [1], corrosion protection of metals, conductive adhesives [2], circuit elements [3, 4] and for sensing applications [5]. Carbon black particles and carbon (nano)fibres are probably the most commonly used conductive components in conductive polymer composites [1, 5–15]. Some photosensitive electrically conductive polymer composites have also been reported. They are composed of a photopolymerizable matrix which allows the material to be lithographically patterned with photons, electrons, ions or X-rays [3, 4]. These so-called conductive photoresists are used either as components of some sensors or for fabrication of electrically conductive micro-components [6, 16–18].

Conductivity of a polymer composite depends strongly on the content of the conductive filler. At small quantities of conductive filler the average distance between adjacent particles is too large and the polymer matrix limits the conductance. At a sufficient amount of filler, the particles get closer forming a conductive network throughout the composite. This filler content is called the percolation threshold. In its vicinity, the conductivity changes drastically even for small changes in concentration of the conductive particles. At higher filler concentrations the conductive paths form an overall spread network and the conductivity changes slightly with further incorporation of the conductive phase [1, 12, 19–22]. The S-shaped conductivity versus filler concentration curve shows an insulator-to-conductor transition.

In production and application processes the content of conductive filler must be low in order to maintain the fabrication advantages and versatility of the host polymer. Hence the percolation threshold should be as small as possible. The influence of the type of conductive filler, the interactions between the conductive and insulating components as well as some properties of the polymer matrix (polarity, viscosity, and degree of crystallization) on the percolation threshold was investigated [1, 9, 12, 15, 19–22].

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Dispersing additives and various preparation processes can be used to disperse the functional filler inside the polymer. However, the literature data on those topics are scarce. The polymer matrix is usually considered to be the insulating host that disconnects conductive paths and allows only the nearest-neighbour tunnelling (percolation-under-tunnelling) [15, 22]. The influence of internal changes in polymer matrix on the electrical transport in percolation systems has not yet been considered.

In the present study, we have studied the resistivity of epoxy-based composite. Photo-sensible epoxy-based polymer (photoresist) was used as the host polymer and conductive CB with small average primary particle size and high specific surface area as functional filler. This way an easy and relatively inexpensive conductive photoresist suitable for simple layer application and for lithographic process was obtained. The electrical conductivity of the prepared composite was studied in relation to the state of dispersion of conductive fillers and to the internal properties of the polymer matrix. These properties are of considerable interest in the standard processing techniques of conductive photoresist, such as spin-coating and lithography.

Due to its excellent chemical and mechanical properties the negative photoresist known under the commercial name SU8 is widely used for MOEMS, MEMS and LIGA applications. Initially developed by IBM for ultra thick layer applications, may give structures with high aspect ratio (>50) [23, 24]. When fully cross-linked, it becomes thermally stable, insoluble in most chemicals, biocompatible and has excellent mechanical strength. Beside the conventional UV lithography it is also used as X-ray definable resist. Our previous study shows even the possibility to form 3D structures by direct proton beam writing [25]. SU8 can be used as sensing material in capacitive micro-machined chemical sensors, with promising discrimination between different volatile organic compounds after appropriate curing [26]. This effect was related to the structural changes in the epoxy-based material, and was followed by IR spectroscopy. The conductive carbon black with small average primary particle size and high specific surface area was applied in the negative-tone photoresist SU8 matrix. Some preliminary results were already published [27].

Experimental

Materials

For this study the SU8-50 photoresist (MicroChem Corporation, USA) was used. SU8 is a solution of glycidyl ether of bisphenol A and photoinitiator (triaryl sulfonium salt) in gamma butyrolactone (GBL) solvent [23]. It gives the highest commercially available epoxide functionality: the average oligomer unit has eight functional groups (epoxy rings) [24].

The commercially available extra conductive carbon black (CB, Evonik-Degussa, Germany) was used as conductive filler. According to the producer data it is a high-structure CB with average primary particle size of 30 nm and extremely large BET surface area (1000 m^2/g).

Two commercially available wetting and dispersing additives (BYK-Chemie GmbH, Germany) were applied to disperse the CB powder. They are high-molecular weight additives denoted here as Dispersant A and Dispersant B. According to the producer data they are similar: high-molecular weight substances with resin-like structure and pigment-affinic groups. Dispersant A is a solution of block copolymer, recommended for application in solvent-based coatings and pigment concentrates where it acts to prevent the reflocculation of pigments. Dispersant B is an acrylate copolymer recommended to use in solvent-free epoxy resins. It stabilizes organic and inorganic pigments in epoxide-functional resins and provides excellent storage stability of pigment concentrates.

Sample preparation

All components of the composite were mixed applying the T18 Ultra-Turrax high-performance disperser (IKA-Werke GmbH, Germany).

Thin layers of dispersions were deposited on a glass substrate and on a silicon wafer for resistivity measurements and for lithographic process, respectively. For applying dispersions on glassy micro slides the Cube applicator (Sheen Instruments Ltd, England) with a 38 μm gap was used, giving a wet film thickness of approximately 38 μm. They were cured on a hot plate (90 °C, 5 min) to evaporate the solvent and to obtain dry layers.

All samples were UV-polymerised by mercury vapour lamp on CANON PLA 501 FA mask aligner. As recommended by the manufacturer the exposure time was 2 min and the energy flux was approximately 200 mJ/cm^2 (http://www.microchem.com/products/pdf/SU8_2-25.pdf). Under such conditions an efficient cross-linking down to the bottom of relatively thick layers is assumed. Finally, samples underwent subsequent thermal treatment for 2 h at 200 °C in air.

The thickness of all composite layers before and after UV-exposure was measured by the profilometre Talysurf (Rank Taylor Hobson Series 2).

The lithography was done on test grade 100 mm silicon substrates. Before the deposition of the composite layer the wafers were treated in O_2/He plasma to improve the adhesion. Commercially available spin coater equipped with a hot plate set to 90 °C was used for layer deposition and subsequent soft bake. Photolithography was performed on the CANON PLA 501 FA mask aligner. A standard test pattern was used to evaluate the photolithography. Prior to the immersion in the MicroChem's SU8 developer, wafers underwent a two-step post exposure bake at 65 and 95 °C for 1 and 2 min, respectively.

Electrical resistivity measurements

The resistance, R, of composite layers was measured by four-terminal method. Electrical contacts were prepared by high-purity aluminium bonding wires (Heraeus, Germany) applying silver electro-conductive adhesive (EpoTek, Epoxy Technology). A constant DC-current source ($I = 1\ \mu$A, $V_{\max} = 100$ V) was used to force the current through the outer contacts of the structure. Voltage drop was measured by an electronic voltmeter (FLUKE 289) on the inner contacts; thus, the problem of contact resistance was omitted. Measurements were done with a 30 s delay to eliminate any charging phenomena. The specific electrical resistivity, ρ, of the composite layer was calculated using the measured voltage drop, V_x, and known current, I, according to the equation:

$$\rho = \frac{V_x}{I} \cdot \frac{W}{L} \cdot T \qquad (1)$$

where, W and L are width and length between inner contacts of the structure, respectively, and T is the thickness of the layer. With this procedure accurate measurements up to $10^8\ \Omega$ (100 V/1 μA) can be accomplished and rough approximations up to $10^{10}\ \Omega$ (100 V/10 nA, the resolution of ampere meter) are made possible. No significant contact resistance and charging phenomena were observed.

Evaluation of the state of dispersion

The state of dispersion of composite layers was evaluated by image analysis of SEM micrographs. They were taken using the field emission microscope Karl Zeiss Supra 35 VP. Public domain software (*ImageJ*) was used for image analysis. It was performed on 30 micrographs, taken on various positions of the same sample at magnifications 10.000 (10 micrographs), 15.000 (10 micrographs) and 20.000 (10 micrographs). The average size distribution of a composite was obtained by averaging these 30 results.

Results and discussion

Electrical resistivity

The DC resistivity of CB-SU8 composites with Dispersant A and B before and after UV-exposure as a function of CB concentration is shown in Fig. 1. All curves show narrow insulator-to-conductor transition where a small increase in CB loading results in a large decrease in resistivity by several orders of magnitude. The percolation concentration, where the resistivity drop is the largest, is at approximately 0.6 vol.% of CB. After UV-exposure the resistivity of all samples decreased; in samples with 0.6 vol.% it dropped for almost five orders of magnitude. This effect is less pronounced at higher CB concentration and becomes negligible at concentrations above 5 vol.%. The resistivity of all samples also depends on the applied dispersing additive. All composites prepared by Dispersant B have lower resistivities than those prepared by Dispersant A. The influence of dispersing additive is larger in unexposed samples than in UV-exposed ones.

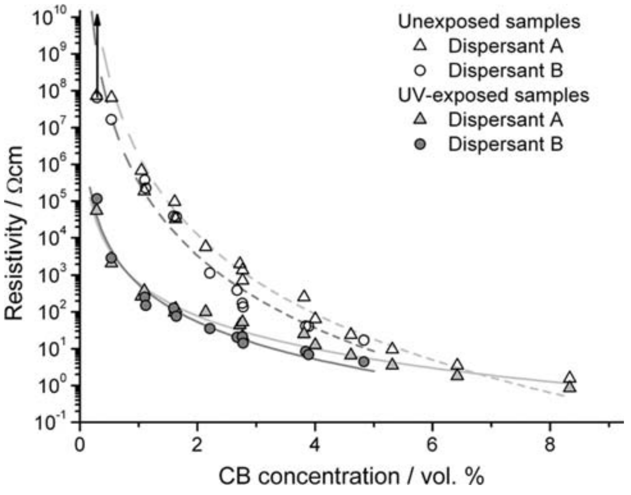

Fig. 1 Resistivity of unexposed (*open signs*) and UV-exposed (*solid signs*) composites in dependence on CB concentration. The samples were prepared using Dispersant A (*triangles*) and Dispersant B (*circles*). The upward oriented arrow indicates that higher resistivity values should be measured for the unexposed sample with 0.3 vol.% of CB. The best polynomial fit to the resistivity values of unexposed and exposed layers are shown by *broken* and *solid lines*, respectively

The change of resistivity, ρ, in the vicinity of insulator–conductor transition beyond the percolation concentration (p_c) can be expressed by the simple statistical percolation model as:

$$\rho \propto (p - p_c)^{-t} \qquad (2)$$

where p is the volume concentration of the conductive filler, p_c is the percolation concentration of the filler, and exponent t determines the scaling behaviour of resistivity [1, 12, 15, 19–22]. The classical theory of lattice percolation assumes that p_c is well-defined minimal concentration of functional filler where inter-particle contacts form at least one conductive path within the composite. For random-packed 3D composites the universal values of $p_c \approx 16$ vol.% and t between 1.5 and 2.1 were predicted [1, 12, 15, 19–22]. However, it is well established that

CB-polymer composites cannot obey nearest-neighbour contact bonding criteria because adjacent CB particles are isolated by polymer. Such particles are likely to be connected by nearest-neighbour tunnelling, giving rise to percolation-like behaviour. This was regarded to be the common model for electrical conductivity of CB-polymer composites. Nevertheless, the quantitative agreement between the classical theory and measured data is not expected for such a system [15, 22].

The percolation concentration p_c of all our composites is 0.6 vol.%—a much lower value than predicted by the classical percolation model, where the mixture-independent mathematical concept gives the universal value of $p_c \approx$ 16 vol.%. However, it is well-established that no universal percolation threshold exists [19] but it depends on several factors including properties of the polymer matrix and of the applied conductive filler. The p_c values reported for CB-polymer composites range from as high as 14% [11], 8 vol.% [1], 2.8 vol.% [20] to as low as 0.75 wt% [7]. These data could be hardly compared while insufficient information about the applied polymer and CB filler were reported. It is well accepted that the value of p_c is diminished by the porosity of the filler and by its irregularity [1, 19]. The influence of polarity, viscosity and degree of crystallization of the polymer was also considered [1, 19].

The measured resistivity versus CB concentration curves shown in Fig. 1 were analysed applying Eq. 2. This way the parameter t for composites prepared with Dispersants A and B before and after UV exposure were obtained. The results are shown in Fig. 2 and Table 1. The $p_c = 0.6$ vol.% was taken into account in all calculations. In UV-exposed samples t is within the theoretical expectations of the standard percolation theory, but in unexposed samples its value is much higher, around 5. The applied dispersing additives slightly influence the t value, more in unexposed samples and less in UV-exposed ones.

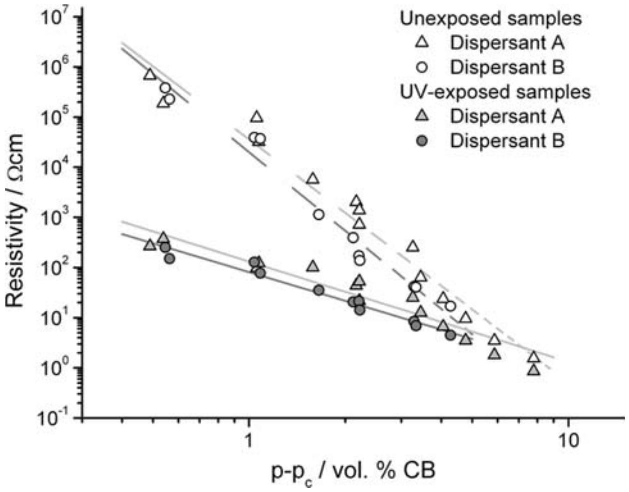

Fig. 2 Application of statistical percolation model (Eq. 2) on unexposed (*open signs*) and UV-exposed (*solid signs*) composites. Data from Fig. 1 were taken into account with $p_c = 0.6$ vol.%. The samples were prepared using Dispersant A (*triangles*) and Dispersant B (*circles*). The allometric fit is represented by *straight lines* and the corresponding t values are given in Table 1

There are literature reports on several reasons why t values are higher than predicted theoretically. The mechanism of electrical transport in such a composite should include an additional conduction mechanism such as multiple percolation or percolation-within-percolation. Such an explanation was given for CB-high-density polyethylene ($t = 3.1$) [20]. It was also reported that in composites with low-structure CB both p_c and t values can differ considerably from the ones predicted by theory, and the t value could be much higher than 2. On the contrary, in composites with high-structure CB filler the t value is expected to approach 2. Therefore, the value of the exponent t was regarded to be the measure of the CB structure: the lower the CB structure, the higher the t value [15]. A low-structure CB consists of aggregates with small number of primary particles. When typical distance between adjacent particles in such a structure is small enough the nearest-neighbour tunnelling occurs and the conductivity is described by high t values. A high-structure CB consists of a grape-like structure of several geometrically connected aggregates. When the inter-particle distances between these macro-particles are small enough the areas of close contact can be formed. In these circumstances the conductivity has a universal percolation-like behaviour and the t value is within the theoretical expectations [15].

The scaling parameter t in CB-SU8 composites decreases from a large value to a theoretical one when samples are UV-exposed (see Table 1), indicating a large change in conductivity mechanism during UV-exposure. According to the above-mentioned literature information [15] an almost close-packed network of small CB aggregates would be expected in unexposed samples ($t \gg 2$) and large CB particles with close contact areas in UV-exposed ones ($t \approx 2$). The possibility of such an explanation was analyzed by size distribution of CB particles in CB-SU8 composite.

Size distribution analysis

Figure 3 shows the size-distribution histograms obtained for unexposed samples with 1.1 and 2.8 vol.% of CB powder. The average particle sizes <d>, obtained from these data are given in corresponding histograms and in Table 1. Typical SEM micrographs applied for this analysis are shown in Fig. 4. The lower concentration (1.1 vol.%) corresponds to just-percolating system with concentration little above p_c whereas at 2.8 vol.% of CB the logarithmic value of composite resistivity is halved. No differences were observed on micrographs of UV-exposed samples, therefore very similar size-distribution graphs and

Table 1 The scaling parameter t and average particle size <*d*> in CB-SU8 composites. Values of t according to statistical percolation model (Eq. 2) were derived by the best fit to data in Fig. 2. Average particle size <*d*> was obtained by image analysis of SEM micrographs at 1.1 and 2.8 vol.% (Figs. 3, 4)

Additive	t (unexposed)	t (UV-exposed)	<*d*> (μm) (1.1 vol.% CB)	<*d*> (μm) (2.8 vol.% CB)
Dispersant A	4.8	2.1	0.266	0.198
Dispersant B	5.2	1.9	0.192	0.152

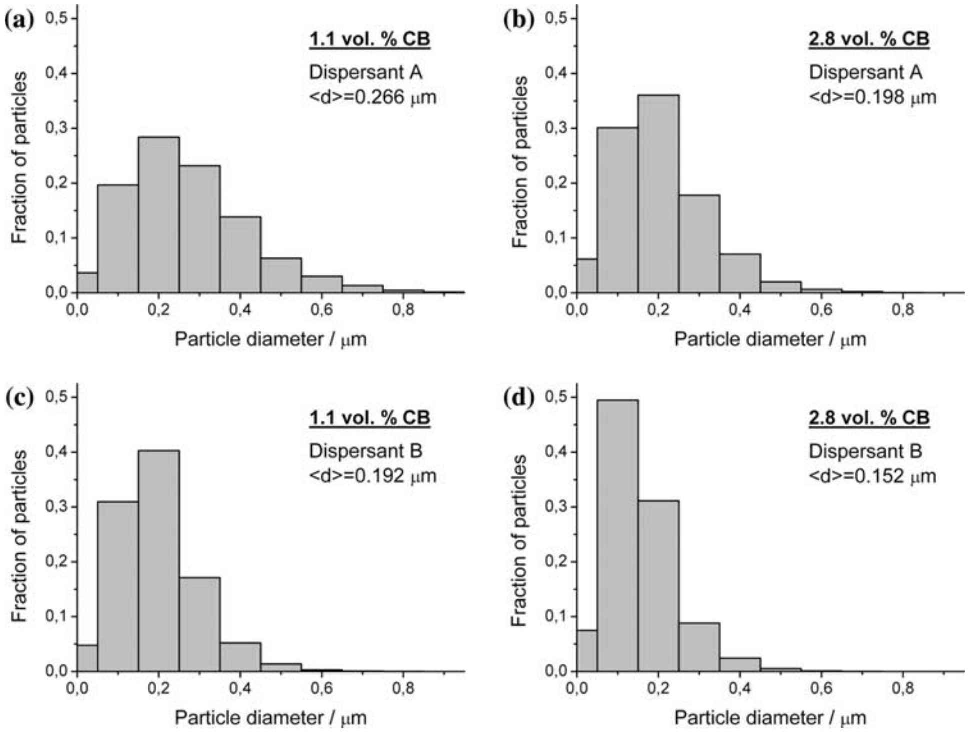

Fig. 3 Size distribution histograms for dry unexposed CB-SU8 composites prepared applying Dispersant A (**a**, **b**) and Dispersant B (**c**, **d**) having 1.1 vol.% (*left*) and 2.8 vol.% of CB (*right*). The corresponding average particle sizes (<*d*>) are given in the legend

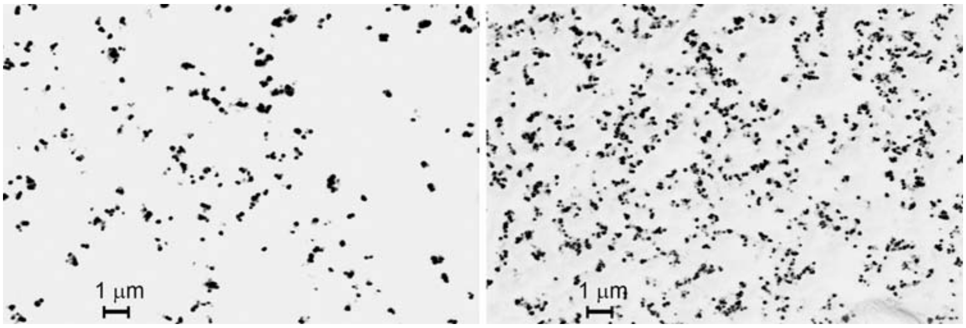

Fig. 4 SEM micrographs of dry unexposed CB-SU8 composites prepared by Dispersant B with 1.1 vol.% (*left*) and 2.8 vol.% of CB (*right*)

<*d*> values were obtained for composites before and after UV-treatment. Composites prepared using dispersant B have size distribution graphs with narrower width and smaller <*d*> than the samples prepared with Dispersant A (see Fig. 3, Table 1). These data show that Dispersant B is more effective that Dispersant A. Larger <*d*> values were obtained at smaller CB concentration and smaller <*d*> values in dispersions with more CB particles due to larger shear stress during mixing. The different resistivity of composites prepared by Dispersants A and B (Fig. 1) most likely reveals the difference in the state of CB dispersion: the larger the <*d*> the higher the resistivity. Therefore, it is reasonable to conclude that better dispersed conductive fillers give rise to smaller resistivity.

The possible reasons why Dispersant B gives better dispersions than Dispersant A could be deduced from the recommendation of the producer. Dispersant B is recommended for coatings with epoxy-resin, whereas Dispersant A is more general and was not designed only for epoxy systems. This specialisation of additive might cause the observed differences.

Image analysis of SEM micrographs shows that the CB particles inside SU8 polymer matrix practically do not change if the sample is UV-exposed. Therefore, the explanation of corresponding *t* values by different structure of CB particles together with different conduction mechanisms reported in the literature could not be applied for the explanation of the conductive behaviour of our system. The same solid composite layer was UV-exposed. During this process CB particles were more-or-less fixed by polymer. The UV-exposure was very short so the sample was practically not heated; therefore larger thermal migration is not feasible. It might be reasonable to attribute the resistivity drop to the changes in the polymer matrix during UV-exposure.

An eight-order of magnitude decrease in resistivity was reported to be obtained during thermal curing of the CB-polymer composite; this effect was contributed to shrinkage of the layer and simultaneous increase of the particle–particle pressure [1]. A negligible shrinkage of UV-exposed layers was observed in our composites while the majority of the solvent has already evaporated during thermal drying. This is a common effect occurring in most of UV-cured polymer systems. Thus, the most important issue is the structural change of the SU8 polymer matrix due to UV-exposure. It was shown previously that the SU8 photoresist layer cross-links during the UV-exposure and additional thermal treatment at 200 °C gives a completely polymerized layer [26]. Such structure is perfectly interconnected by 3D cross-links so that practically no open links between adjacent units exists. We could hypothesize that such structure is promoting the charge transport across composite better than the opened structure of the unpolymerized (unexposed) matrix. The effect is very important at small CB concentration and becomes almost negligible at higher. We may conclude that in composites well-above the percolation threshold the conductivity of CB particles is predominant factor in defining conductivity. When conductive network is formed (i.e., well above p_c) the composite becomes poorly sensitive to further increase in CB concentration.

Similar influence of the polymer matrix seems to be obtained by the two predominant methods of polymer modification, grafting and cross-linking [28–30]. Grafting is chemical bonding of polymers onto nanoparticles which considerably influences the electrical properties of the material. Further research is needed in this direction.

The applicability of the conductive photoresist

The incorporation of functional filler should preserve the matrix properties as much as possible. For these reasons the applicability of our composites for photolithography was evaluated. The thickness of layers prepared by spin-coating depends on the spin speed of the coater and on the viscosity of the composite which is influenced by the CB content. The dependence of thickness on the speed of the spin-coater for two composites with different formulation (0.2 and 1.1 vol.%) after soft bake at 90 °C is shown in Fig. 5. For comparison only, the curve of pure SU8-2 photoresist used for target thicknesses of about 2 µm is also shown. Differences in thickness versus spin speed curves display different viscosities. Using the producer data it is possible to conclude that the viscosity of composite with 0.2 vol.% of CB corresponds to that of the SU8-10 (1050 cSt) and composite with 1.1 vol.% of CB to the viscosity of the SU8-5 (290 cSt) since the thickness versus spin speed curves almost overlap (http://www.microchem.com/products/pdf/SU8_2-25.pdf).

The addition of CB in concentrations below 1.1 vol.% does not destroy the resolution of the photolithography as checked by the standard test pattern with line widths from 2 to 10 µm for composites with 0.2 and 1.1 vol.% of CB (Fig. 6). The measured difference in line widths of resist and mask lines depend on the exposure time (UV-exposure dose) as depicted in Fig. 7. For the sample with 1.1 vol.% of CB the correct exposure times have to be longer for about 60% compared to that with 0.2 vol.%. For such low CB concentrations the exposure time is comparable to that of the pure SU8 resist, which is about 30 s on the mask aligner

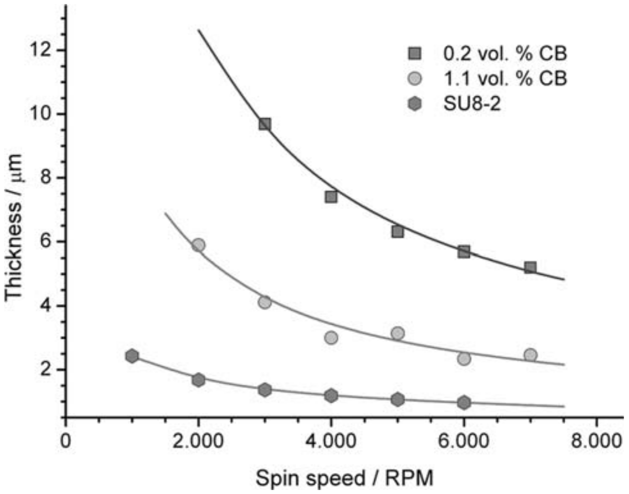

Fig. 5 Thickness of spin-coated CB-SU8 composite layers as a function of spin speed. Composites were prepared with Dispersant A (*squares* 0.2 vol.% CB, *spheres* 1.1 vol.% CB). The result obtained applying the pure SU8-2 resist is shown for comparison (*hexagons*)

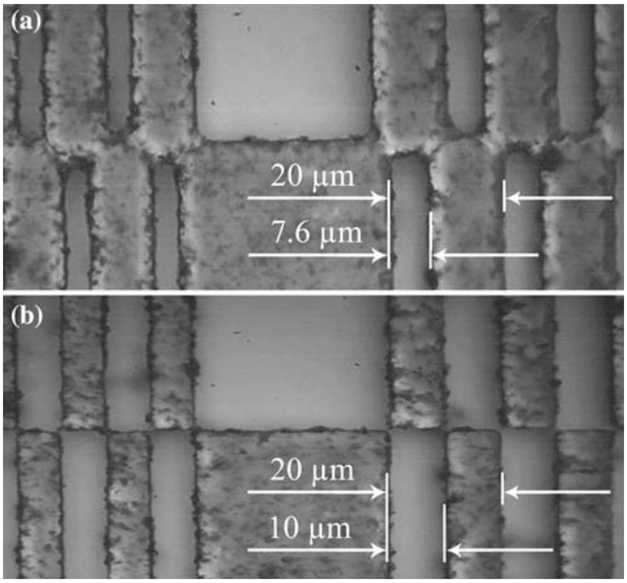

Fig. 6 Photomicrographs of standard photolithographic test patterns prepared with CB-SU8 composite layer containing 0.2 vol.% of CB applying Dispersant A. The thickness of the layer was 6.3 µm. The patterns were obtained by exposure times of 76 s (**a**) and 30 s (**b**)

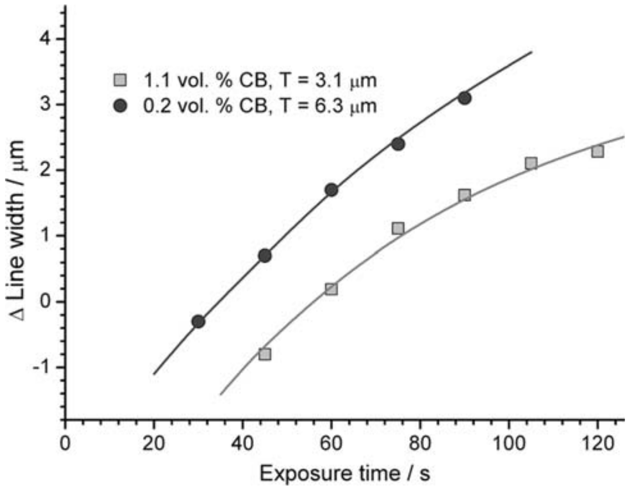

Fig. 7 Line width difference as a function of exposure time for CB-SU8 composites prepared with 0.2 vol.% CB (*circles*) and 1.1 vol.% CB (*squares*). See also Fig. 6

used. According to the manufacturer data (http://www.microchem.com/products/pdf/SU8_2-25.pdf) the energy flux needed for photopolymerization is approximately 200 mJ/cm^2. The thickness of the sample with higher CB concentration ($T_{1.1} = 3.1$ µm) is only one half of the thickness of the sample with 0.2 vol.% ($T_{0.2} = 6.3$ µm). On the photo-micrograph (Fig. 6) rather sharp edges with some irregularities for correctly exposed sample can be seen. With increasing CB content in the photoresist matrix the exposure time increases and the resolution of test lines decreases as the edges become rough.

Conclusions

The objective of our research was to prepare conductive photoresist with acceptable photolithographic properties. The near-UV photosensitive epoxy-based polymer (negative-tone photoresist) was used as the insulating matrix and the extra-conductive carbon black as the conductive filler. High conductivity of composite with preserved properties of the matrix was desired. The research was therefore divided in two basic tasks: the study of the electrical properties of composite as a function of filler concentration and the testing of prepared composites for photolithography. The information obtained by both tasks provides the necessary data for the smallest filler concentration and the preparation condition for a good conductive photoresist.

The well-known S-shape of the resistivity versus concentration curve of the composite was obtained. The resistivity decreased after UV-exposure, but the size of this effect depends on the concentration of the conductive filler. The most prominent change occurs at concentrations near the percolation threshold (~0.6 vol.%) where the resistivity drops for almost five orders of magnitude. The effect is reduced at higher filler concentration and becomes almost insignificant at 5 vol.%. The whole effect was attributed to the changes in the polymer matrix going from unpolymerized to completely polymerized state upon UV-exposure. The chemical structure of a fully polymerized matrix has no open links between adjacent oligomer units, which most likely helps in promoting electrical conductivity throughout the composite. This effect is important at low filler concentration and becomes negligible at higher where the resistivity is likely controlled only by the conductive paths formed by the filler.

Better dispersed filler particles give rise to smaller resistivity. This effect is clearly recognisable in unexposed composites at concentrations below 5 vol.% and diminished considerably after the UV-exposure. The state of dispersion of conductive particles, therefore, appears to be more important when the applied polymer matrix is poorly cross-linked.

The presence of filler particles changes the viscosity of the host material. The spin-coating method still remains reliable for composites with filler concentrations of up to at least 1.1 vol.%. The addition of fillers up to 1.1 vol.% does not reduce the resolution of photolithography.

It appears that the large change of resistivity observed at concentration slightly above the percolation threshold after UV-exposure of the composite cannot be explained within the comprehensive percolation-tunnelling model proposed in the literature. The polymer undergoes important structural changes during UV-exposure and it is very likely that these changes play an important role. However, many questions are still open, especially the chemical properties of the interface between polymer matrix and conductive fillers as well as the influence of dispersing additive on this surface. Further research in these directions will give better insight on the effects and resistivity changes shown here.

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