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# Conductivity enhancement of silver filled polymer composites through electric field alignment

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ABSTRACT

We show how an alternating electric field can be used to align silver micron or sub-micron sized particles into microscopic wires in diverse polymer matrices based on the dielectrophoretic effect. The electric field is set by an electrode pair and the wires form conductive pathways through the matrix, bridging these electrodes electrically. The matrix is cured after alignment, locking wires in permanent pathways within the polymer. The wires are then characterized by ac impedance spectroscopy. The alignment can take place either in-plane or out-of-plane, and yields a directional conductivity in the alignment direction parallel to the electric field lines. The samples can be centimeters wide containing thousands of wires in parallel, but even an individual wire can be grown and controlled. The initial mixture contains less than 1 vol.% of silver and is an electrical insulator. The bulk conductivity enhancement, due to the alignment, may be 5 orders of magnitude, typically from $1 \times 10^{-5}$ S/m to 1 S/m as the particle alignment converts the sample conductivity from polymer dominated to silver dominated. For the aligned isolated silver wires, the jump in conductivity, confined to the volume filled by the wire can be seen to be as high as 9–10 orders of magnitude, resulting in conductivities as high as $1 \times 10^{5}$ S/m, thus approaching those of pure metal. This technique offers new ways on how e.g. conducting polymer composites and conducting glues could be produced.



## 1. Introduction

By applying an alternating electric field between a suitable set-up of two or more electrodes, conductive particles dispersed in a fluidlike polymer or polymer precursor matrix can be manipulated to align into thin conductive strings, bridging the electrodes electrically even at particle filler ratios tenfolds below the percolation limit. The driving mechanism behind such a field alignment is dielectrophoresis (DEP) by which a polarizable, but charge-neutral particle feels a net force parallel to the electric field lines [1]. The only requirements for an existing DEP force are the field gradient be non-zero as well as must the particles and matrix have non-equal electric permittivities. If the particle is more polarizable than the dispersing matrix, the particles will move in the direction of increasing field strengths, and so the particles will start aligning into wires at the electrodes since the DEP force points towards the electrode edges [2]. Once the wires are complete, the matrix can be cured rendering solid anisotropic material.

In industries such as electronics packaging, electric interconnections are traditionally established by soldering that ensures high electronic contact [3]. However, such a soldering also involves environmental unfriendly metals as well as high temperatures yielding mechanical stress, factors which are both unwanted [4]. Therefore, electrical conductive adhesives are experiencing increased attention [3,5], and potential applications include fills for semiconductor packaging [5], interconnection of solar cells [6] as well as in making electro-mechanical connections in touch screens [5,7]. Commercially available isotropic conductive adhesives are generally based on silver particle loadings of as high as 70–90 wt.% in an insulating polymer [3,5,8–10]. The high silver particle loading, well above the percolation limit, ensures conductive paths in all directions within the adhesive due to unavoidable particle–particle contacts. However, such high silver loadings are cost inefficient, suppress polymer properties such as adhesion to e.g. metal surfaces [3,11], optical transparency, as well as generating an often unavoidable macrophase separation [12]. Printing of pastes becomes difficult as high loadings of nanomaterials cause a drastic increase in viscosity of the paste [11]. Due to the reduced optical transparency caused by the filler particles, curing of the matrix cannot be performed by the well-controlled UV process [13], but must be performed by thermal methods or other time- and cost inefficient methods [11]. Orienting graphite sheets, dispersed in a polymer resin, by a dc electric field has proven to increase the

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optical transparency significantly [14]. From a practical working point of view, UV curing is a superior principle to control the curing of the polymer. Thus, a semitransparent polymer composite, due to a significantly reduced particle loading, that also conducts will be useful in many application areas.

One method to produce conductive adhesives at low filler ratios consists of using specific high aspect ratio particles as fillers, as this may result in a lower percolation limit [5,15]. The particles can be chosen long enough to reach from one electrode to the next, yielding anisotropic conductive adhesives [15,16]. However, this may impose restrictions on the maximal distance between the electrodes, as well as provide an uncertain method for making conductive bridges as the particle may not hit both electrodes.

It would therefore be an advantage if the conductive adhesives and similar composites were constructed by a much lower particle filler ratio by aligning small particles into thin, highly conductive wires. Such an alignment might be performed either across the materials thickness or parallel to its surface, all depending on the final product purpose. This would also enable a directional conductivity, if desired, yielding conductivity only in the direction of interest as a curing of the polymer locks the aligned wires in a stable configuration [17]. Anisotropic structuring of carbon based filler particles, although of high volume fractions, generated by different alignment methods, has previously shown to enhance the adhesives mechanical properties [14,16] as well as the thermal conductivities [18] along the direction of alignment.

Electric field alignment studies of various carbon filled polymers have previously been carried out, in some also with conductive adhesive applications in mind. Materials used for these purposes have been carbon nano cones [17,19], carbon nano tubes [20–22] carbon black [7,23,24], and carbon fibers [25]. These materials allow bulk conductivities up to $10^{-1}$ S/m [25]. Elsewhere, studies of dielectrophoretic growth of metallic wires [26], electric field alignment of gold nano particles [27], silver nanowires [28] as well as magnetic alignment of nickel particles [29] have also been performed, but not with micro or nano-sized silver particles as fillers with the purpose of high conductive aligned wires with conductive adhesives applications in mind.

In this paper we have used silver particles as fillers with the purpose of making high conductive adhesives at low particle filler loadings. We show that the particle loading, resulting in conducting wires, can be as low as ten times below the percolation limit when the polymer matrix is subject to an alternating electric field, and we seek a method for making conductive adhesives at minimal loading ratios and cost. The silver particles are aligned into thin conductive wires from an initially isotropic and low fraction polymer with no macroscopic electric conductive properties, yielding anisotropic conductivity and a significant jump in conductivity in the direction of the applied electric field. We present results of electric field alignment of silver particles of micro or sub-micro particle size, both in terms of individual wires as well as larger bulk samples, consisting of thousands of wires in parallel, and investigate their general electrical properties. In total, we show how conductive adhesives, consisting of silver aligned wires, can be constructed from a very low particle fraction, enhancing the polymer properties. The results of this study have implications on how conductive adhesives are produced, with implications in areas such as solar cell interconnections, conductive, transparent films, sensor applications and electronics packaging.

## 2. Materials and methods

### 2.1. Materials

The silver particles used in this study were commercially available flakes (>99.9%, <10 μm) provided by Sigma–Aldrich. Two different adhesive polymers were used for dispersing the silver particles. A two component thermoset polymer containing Araldite AY 105-1 epoxy resin (Huntsman Advanced Materials) with Ren HY 5160 or HY 5162 amine hardener (Vantico AG), and a UV-curable Dymax 3094 acrylated urethane based polymer (Dymax Corp.). Both choices of polymers were of technical quality and supplied by Lindberg & Lund (Norway). Mixing of the silver particles with the polymers was performed by a 120 rpm stirring at room temperature for 20 min or more. For alignment studies the particle concentrations were ranging from 0.1 to 0.5 vol.% which is well below the expected value for the percolation limit.

### 2.2. Electric field alignment

The alignment procedure and used alignment geometries are illustrated in Fig. 1. For the generation of single wires, the silver matrix was placed to form a uniform, thin layer on top of tip-like electrodes (Fig. 1A). The thickness and width of these electrodes were 100 nm and 3 μm, respectively, and the tip-tip distance was in the range of 10–100 μm. For larger samples with multiple wires, the alignment of the silver particles was either performed horizontally (in-plane alignment; Fig. 1B) by placing the matrix on a glass microscope slide with prefabricated electrode fingers under optical microscopy observations, or vertically between a top and bottom metallic electrode (out-of-plane configuration: Fig. 1C), as described in more detail in earlier works [19]. The distances between the alignment electrodes were in the range of 50–1000 μm in both geometries. In all our experiments, the silver–polymer dispersion was placed onto the electrode-set-up immediately after the mixing. In this procedure there was no time for significant particle precipitation.

Before any alignment procedure to be initiated to a matrix, its resistance through the electrodes was measured by a Fluke 179 multimeter. The ac electric field needed for particle alignment was provided by a B&K Precision function generator combined with a Model 2210 amplifier (Trek Inc.). The signal frequency used was 1 kHz sinusoidal, and electric field strengths (rms values) were typically of 1–4 kV/cm, lasting from 10 s up to several minutes. The alignment of the silver particles was monitored by optical microscopy, and the enhancement of the matrix conductivity was studied by dc and ac electrical measurements. Curing of Araldite matrix was performed by moderate heating at 60 °C for 1 h. Curing of the Dymax matrix was performed by UV light using Dymax Blue Wave 200 UV source.

### 2.3. Electrical characterization

The detection of the conductivity enhancement was observed by the sudden drop of resistance, measured by a Fluke 179 multimeter. The process was monitored in an optical microscope (Nikon Optiphot), with possibilities of continuous image recording (DinoCapture 2.0). The silver conductivity data estimates, both for the single wires as for the bulk samples, were performed using Ohm's law for volume conductors. For the single wire conductivity estimates, a most correct value of the volume of each wire was achieved based on the optical images of the completely connected wires. The wires were modeled as massive cylinders with a best estimate of the diameter obtained based on the optical data. For the in-plane aligned conductive sheets, the factor to be determined for conductivity estimates was the vertical thickness of the film, which was set to 50 μm.

The impedance analysis of the aligned wires was performed after the samples had been cured, locking the wires in a conducting configuration, suitable for e.g. transportation and further electric characterization. In that respect, the dc electric conductivity study was completed with an ac electric impedance spectroscopy for the

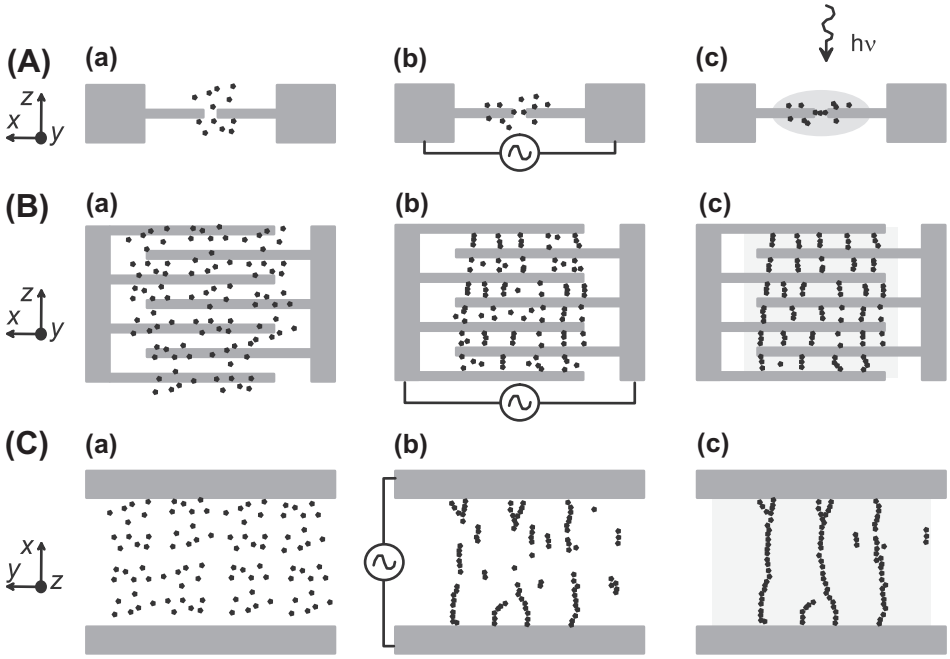


**Fig. 1.** Schematics of the set-ups used for preparations of conductive adhesives with aligned silver wires. (A) Refers to the single wire generation from an isotropic, (a) 0.15 vol.% silver mixture through, (b) electric field alignment and (c) curing of polymer by UV light. (B) Refers to in-plane alignment, creating conductive sheets through the same procedure. (C) Refers to out-of-plane alignment.

single wires as well as for the in-plane macroscopic aligned sheets. For the purpose of such measurements, a Solartron SI 1260 combined with a Solartron 1294 interface was used in a two-terminal configuration, with applied frequencies ranging from 100 Hz to 1 MHz and with a measuring voltage of 30 mV (rms).

## 3. Results and discussion

### 3.1. Alignment of silver particles – single wires

The enclosed multimedia video clip, in Fig. 2, shows the time evolution of the formation of a silver single wire through the dielectrophoresis process. When an alternating electric field was subjected to the particle dispersions, the silver particles started to align into clusters, forming a wire parallel to the electric field line. Fig. 3a and b show the final result, in more detail, of two single wires after UV curing of the polymer matrix. The distances between the electrode tips of these two single wires were 100 μm and 10 μm, respectively. As seen from the video clip, it took less than 25 s to establish a conducting wire from the initially high resistive low filler silver adhesive by applying an electric field of 3–4 kV/cm. Before the alignment took place, the resistance of the silver mixture was of the order of tens of MΩ, and this initial value was typically seen for all the non-aligned and non-cured mixtures in our study. The corresponding conductivity of the isotropic, non-aligned matrix was estimated to be of the order of $1 \times 10^{-5}$ S/m or lower and is attributed to the polymer precursor. The volume fractions of silver of the wire in the video clip as well as the wires in Fig. 3, were 0.15%, which is of the order of tens lower than the percolation limit of disk shaped particles [30].

Starting from an initially, electrically insulating matrix, the silver particle alignment induced a large jump in conductivity as the wire formation was complete. The silver wire shown in the video clip, had a resistance of 451 Ω after the alignment process and this value remained constant, at least within ± 10 Ω when the polymer was UV cured, locking the particles in a confined position. Furthermore, we observed the conductive character of the aligned wires in this study to remain stable for at least 6 months after curing. The structure of the wire is seen to consist of aligned particles or assemblies of particles. The other single wires in this study had resistance values ranging from 284 Ω to 577 Ω after alignment, depending on their length and thickness.

Based on the optical micrographs of the single wires as well as the maximal size of the silver particles that were aligned, a best estimate of the dc conductivity, $\sigma$ of the single wires was made using

$$\sigma = \frac{d}{AR}, \tag{1}$$

where $d$ is the length of the wire, $A$ the cross-sectional, circular area and $R$ is the measured resistance. The wire in the video clip, had an estimated conductivity of $0.9 \times 10^5$ S/m, obtained by using an approximated value of the wire diameter of 1 μm. The other single wires in the study had conductivity values in the range of ca. $4 \times 10^4$–$3 \times 10^5$ S/m. We observed that a minority of the aligned single wires were not conducting after the alignment was complete although the wires appeared to be a connected string as seen by the optical characterization.

The conductivity values of the aligned single silver wires did not reach the same level as the bulk silver material, but were two to three orders of magnitude lower. This observed difference is likely to be due to the contact resistance between the particles [30,31], and is thought to be a general feature for conductors made of assembled or aligned particles, as well as for random networks generated from high filler loadings. In the study by Untereker et al. [30], the conductivity of various particle filled polymers never exceeded 1% of that of the pure particle material itself, and this is similar to our findings for the single wires consisting of silver particles.

This also explains naturally that a minority of the aligned single wires did not conduct well, even though they appeared to be continuously connected as observed through optical microscopy inspection. A thicker layer of e.g. polymer at some point along

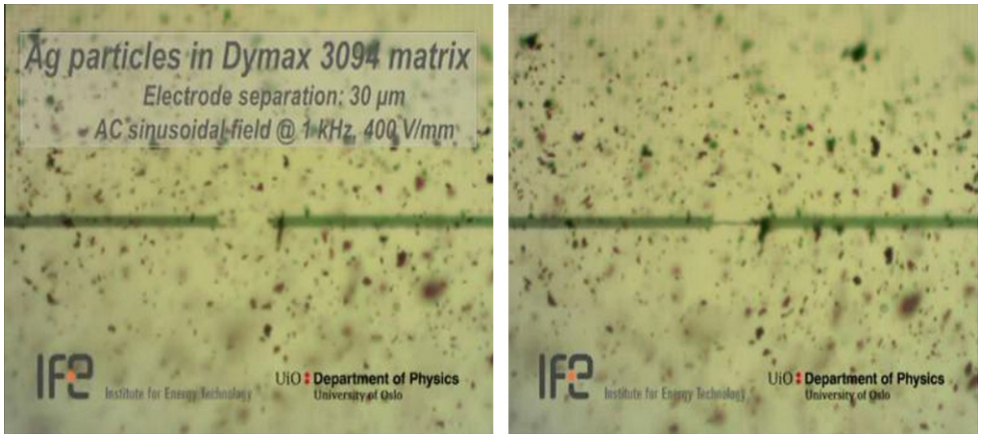


**Fig. 2.** Snapshots of a video clip of silver particles alignment, resulting in a single wire. The left image was taken before, whereas the right after 15 s of alignment. The distance between the electrode tips were 30 μm, the volume filler fraction 0.15%. The electric field strength was 4 kV/cm.

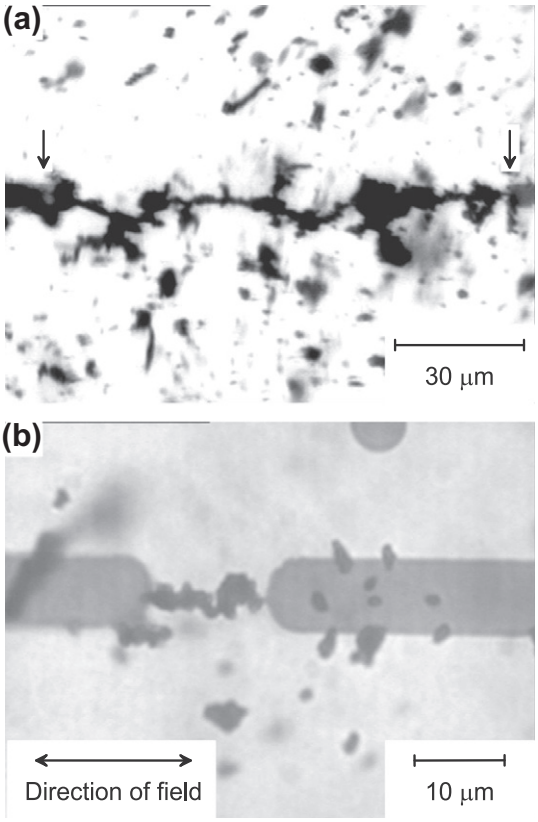


**Fig. 3.** Optical micrographs of single silver wires induced by electric field alignment. Each image was taken in the transmission geometry. The volume fraction of silver was 0.15% in both: (a) 100 μm wire, and in (b) 10 μm wire and the applied electric field was 1–2 kV/cm during the alignment process. The arrows in (a) mark the electrode tips and the arrow in (b) indicates the direction of the applied field. The two electrode tips are seen in the left and right end of the two images, respectively.

the wire due to a geometrically unfortunate configuration of contact points between adjacent particles may switch the wire appearance from high conductive, particle–particle dominated to a high impedivity polymer dominated nature. If the tunneling distance exceeds about 2 nm, the particle–particle contact essentially forms an insulator [32]. The dependence of the tunneling distance effectively makes the wires sensitive to deformations, and so this observation could be useful in sensor applications.

### 3.2. Aligned sheets

Fig. 4a shows an example of a conductive sheet, generated by in-plane alignment of a 0.5 vol.% silver particle mixture placed on top of a grid of electrode fingers where the neighboring fingers were of opposite polarity. The resistance through the alignment electrodes was monitored. Before alignment the resistance was not measurable (>50 MΩ) with our instrument but after alignment and curing it was 50 Ω. The electrode area was 1 $cm^2$ and the electrode spacing 50 μm and the film thickness 10 μm, which means conductivity of at least 1 S/m. These films are translucent. Fig. 4b shows a photo of the macroscopic in-plane aligned sample in Fig. 4a.

Fig. 5 illustrates the process of making conductive sheets, consisting of multiple aligned wires in parallel, from non-conductive, low filler ratio dispersions. This example concerns much longer wires than those shown in Fig. 4a. The volume fraction of the silver dispersion shown in Fig. 5 was 0.4% and the alignment was performed in-plane as illustrated in Fig. 1B. The initial resistance of the mixture was in the order of tens of MΩs, making it effectively an insulator. After about 1 min of electric field alignment, multiple silver wires formed in parallel between the two alignment electrodes that were separated at a distance of 1 mm. The resulting resistance dropped abruptly to 410 Ω at the instant when the wires were bridging the electrodes, remaining stable as long as the electric field was present. The conductivity of the polymer sheet after alignment was found to be of the order of 50 S/m. The estimated height of the polymer film was 50 μm, corresponding to at least 5 times the typical wire thicknesses. The conductive sheets that were produced by means of particle alignment in this study, had a final resistance between 85 Ω and 420 Ω, depending on the silver particle filler ratio and width of sample as well as the distance between the alignment electrodes. There was a trend that higher filler ratios gave lower resistance values after alignment, following a linear or linear-like increase with initial particle concentration. This is in accordance with our expectations that higher filler ratios enabled more wires to be formed in parallel.

### 3.3. Aligned bulk

Fig. 6 shows a microscopy image of out-of-plane aligned silver particles. The electrode distance was 300 μm and the particle filler loading was 0.5 vol.% In a similar way as in the in-plane configurations, the slightly asymmetric silver particles were aligned along the electric field, but also with respect to each other, forming assemblies that eventually formed multiple wires in parallel. In

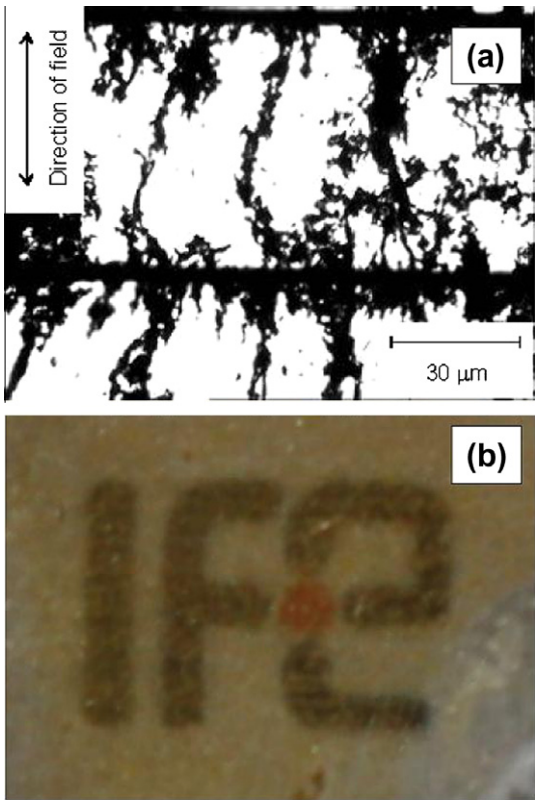


**Fig. 4.** Top view optical images of aligned particles (a). Silver particle loading in the polymer was 0.5 vol.% The horizontal, black bars are the alignment electrodes. Top view photography (b) of the in-plane aligned sample in (a). The text visible through the sample was about 0.5 cm wide. Both images were taken in the transmission geometry.

this kind of samples, the electrical resistance over the sample behaved largely as in the samples aligned in-plane.

In overall, the conductivity enhancement due to the field alignment was of the order of 5–6 orders of magnitude for the in-plane as well as the out-of-plane macroscopic multiple aligned wire mixtures. Also, these macroscopic samples did not show the same critical dependence of the conductivity on the aligned particle configuration as the single wires. This is due to the fact that the macroscopic samples contained multiple wires in parallel, ensuring fully connected wires within the polymer matrix.

### 3.4. Electrical characterization

Fig. 7 shows the ac impedance spectroscopy data of the single wires, the in-plane conductive sheets consisting of multiple aligned wires, as well as the isotropic, non-aligned samples. The impedance data clearly show that the single wires as well as the larger sheets are predominantly resistive at all frequencies between 100 Hz and 1 MHz. This means that there is not just a dc conductivity enhancement present due to the electric field alignment, but also an ac conductivity enhancement for frequencies up to at least 1 MHz, although the conductivity gain was observed to be largest at the low frequencies. The non-aligned samples showed predominantly capacitive nature with a low ac conductivity that appeared to be proportional to the applied signal frequency as the measured impedance was observed to be inversely related to the frequency. At all the measurement frequencies, the aligned wires, both single as well as multiple, had a lower impedance than the non-aligned silver dispersion matrix.

The high electric admittivities of the aligned mixtures are expected to be due to a close particle assembly of silver particles, meaning that they are in physical contact with only a thin polymer or oxide coating between the particles or clusters of particles. The contributing factors to the admittivity are then the particles themselves as well as the contact admittance between individual particles. The thin layers of lubricants, polymer wetting or oxide layers dominate the impedance of the wires, still being of resistive nature, seen from the ac impedance measurements in Fig. 7, where the impedance was observed not to change with frequency. At sufficiently high frequencies, the capacitive conductivity of the polymer or non-aligned silver mixtures can be expected to dominate. As seen from Fig. 7, an extrapolation of the impedance data of the isotropic mixtures, yield lower impedance than the aligned mixtures above a certain frequency value. This was as expected and can be explained by an inspection of the expression of complex admittance $Y$, describing two conductive mechanisms, dc conductive (silver) and capacitive (polymer) in parallel.

$$Y = G + j\omega C = \frac{A}{d}(\sigma + j\omega\varepsilon), \tag{2}$$

where $G$ is conductance, $\omega$ ac signal angular frequency, $C$ capacitance, and $\varepsilon$ is electrical permittivity of the polymer. This implies dc resistive dominance of the mixture given that the measuring frequency satisfies $f < \frac{\sigma}{2\pi\varepsilon}$, where $\omega = 2\pi f$, and $f$ is signal frequency.

The results in this study show how a large increase in conductivity of silver particle filled polymer dispersion can be established

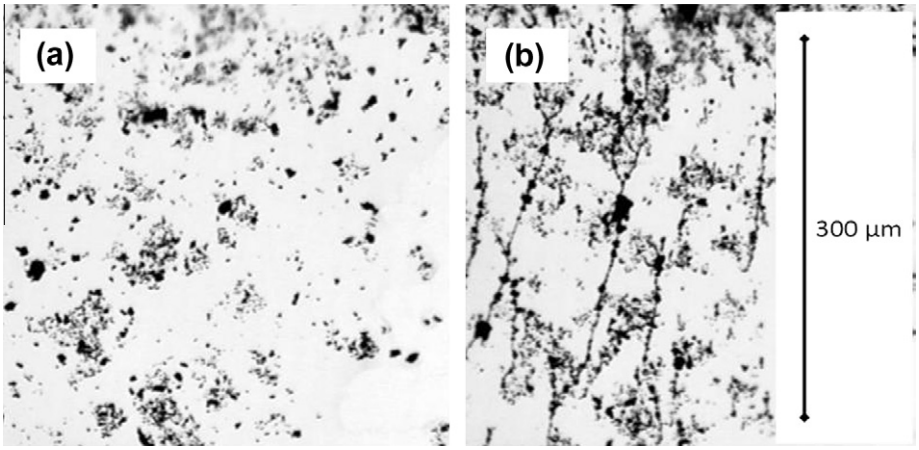


**Fig. 5.** Optical micrographs of aligned silver particles over an electrode distance of 1 mm. 0.25% vol. (a) Isotropic and non-conducting mixture. (b) Mixture after electric field alignment of 1 min, generating conductive wires in-plane. Both images were taken in the transmission geometry.

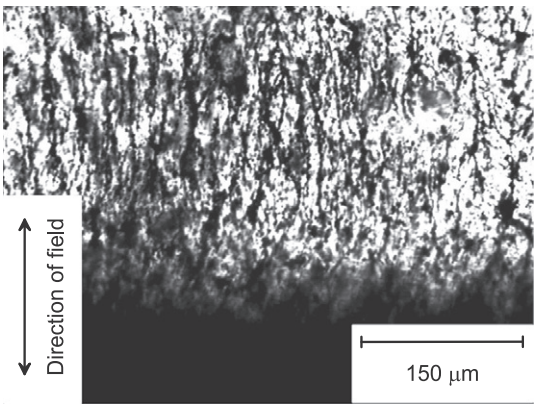


**Fig. 6.** Side view optical images of aligned silver particles. The particle loading in the polymer was 0.5 vol.%

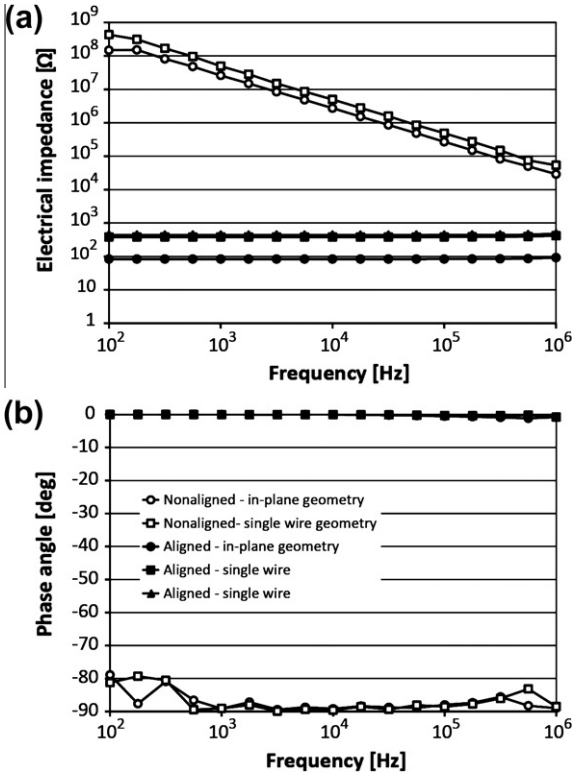


**Fig. 7.** (a) Absolute value of the electrical impedance, *Z*, and (b) phase angle of the studied silver filled adhesives. The open markers correspond to the non-aligned samples, whereas the filled markers correspond to the aligned 0.15 vol.% single wires as well as a 0.4% aligned in-plane, macroscopic sheet (filled circle).

within a short time by applying an ac electric field to the polymer mixture. The conductivity enhancement obtained due to the particle alignment was found to be of the order of 5–6 orders of magnitude for macroscopic samples, containing silver wires either in-plane or out-of-plane. For the single wires isolated, the enhancement is naturally much higher as this local volume in the polymer is converted from low-particle into a region of densely aligned silver particles. The jump in conductivity upon alignment may therefore be as high as 9–10 orders of magnitude, representing the large transition from the matrix dominated conductance ($\sigma \sim 1 \times 10^{-5}$ S/m) to the packed silver dominated conductance ($\sigma \sim 1 \times 10^{5}$ S/m).

The conductivity values that were obtained in this study are comparable to (single wires, Fig 3) or lower (macroscopic sample, Fig 4–6) than those found in studies of isotropic conductive adhesives where silver loadings between 70 wt.% and 90 wt.% were used [3,5,10]. The values should be interpreted as distributions rather than exact numbers due to the uncertainty in e.g. our estimates of wire diameter and film sheet height. We expect the various distributions to be within one order of magnitude. The conductivities of the macroscopic samples are naturally lower than those of the corresponding high filler loading isotropic adhesives as there is much more polymer surrounding the conductive wires, but of the order of values found in attempts in making conductive films [33]. The electrical conductivity of the aligned wires is expected to depend on that of the silver material itself, the constricted cross-sectional area between the particles, as well as surface properties of the silver particles, such as coatings of oxides or lubricants polymer properties, such as the tunneling resistance of the polymer coated interface between the particles [11,13,31,32].

The video clip of the single wire formation shows the rapid formation of a single silver wire through electric field alignment. The time needed to form fully connected wires generally depended on the filler loading as well as the distance between the alignment electrodes. If the filler ratio was increased, the time required to create conductive networks dropped accordingly as the neighboring particles then were at closer distances in the initial, isotropic mixture. The rapid processing speed in making aligned conductive adhesives makes this technique interesting for industrial applications and purposes. The fact that the particle alignment is driven by the electric field, and not the passing current, means that conductive wires can be produced without being in physical contact with the alignment electrodes. This might also enable higher processing speed, as the alignment of the particles can be performed at a distance, preventing the adhesive from curing onto the setup. The reduced viscosity of the adhesives in our study, due to the drastic reduction of filler loadings [11], makes it easier to implement them in low-cost jet dispensed printing applications [34,35].

Compared with other conductive adhesive products, our study leads to a generic and simplified, low-cost approach that contains fundamental benefits: Apart from the apparent cost and weight reduction, due to the massive reduction of silver as well as a simplified curing mechanism (through UV light), the new conductive adhesives can be expected to show lower stress, enhanced mechanical properties as well as significant increase in optical transparency, beneficial for transparent conductors and photovoltaics. This is especially promising as the printed alignment electrodes as well as the wires themselves were not visible to the naked eye, yet being of macroscopic lengths. The length of the aligned wires was up to 1 mm, both for the in-plane as well as for the out-of-plane configuration, but is not believed to be restricted to that and hence can be made longer. Combined with the observations that the silver wires remained stable conductors over time periods longer than 6 months, the technique used in our study is promising for new conductive adhesives products that require stable, high conductive properties over lengths beyond the microscopic lab scales.

## 4. Conclusion

In this study we have shown how an alternating electric field can be used in order to align micron- and sub-micron sized silver particles into thin, conductive wires when dispersed in a commercial polymer matrix which can be adhesive. This induces a markedly jump in electrical conductivity in the composite. Our method enables new approaches on how conductive adhesives and composites are generated, as the underlying physics is generic and can have implications in many application areas. For increas-

ing conductivity further still, the nature of the contact resistance should be studied further as it is the factor preventing the silver wires from reaching conductivity values of the bulk material itself. Reducing the contact resistance, for instance by enabling metallurgical joints, would be beneficial for the preparation of high conductive adhesive composites.

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## Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.compscitech.2012.07.011.

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