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Achieving electrical conductive tracks by laser treatment of non-conductive polypropylene / polycarbonate blends filled with MWCNTs

Marco Liebscher[1], Beate Krause[1], Petra Pötschke[1*]
Andrea Barz[2], Jens Bliedtner[2], Michael Möhwald[2], Alexander Letzsch[2]

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1 – Marco Liebscher, Dr. Beate Krause, Dr. Petra Pötschke, Leibniz-Institut für Polymerforschung Dresden e.V. (Leibniz Institute of Polymer Research Dresden, IPF), Hohe Str. 6, D-01069 Dresden, Germany)
E-mail: poe@ipfdd.de

2 – Dr. Andrea Barz, Prof. Dr. Jens Bliedtner, Michael Möhwald, Alexander Letzsch
Ernst-Abbe-Fachhochschule Jena - University of Applied Sciences Jena, Carl-Zeiss-Promenade 2, D-07745 Jena, Germany

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Electrical non-conductive polymer blends consisting of a polypropylene (PP) matrix and dispersed particles of polycarbonate (PC) were melt compounded under filling with 3 wt.% multiwalled carbon nanotubes (MWCNTs) and processed into plates by injection molding. The morphological analysis confirmed the desired complete selective localization of the MWCNTs in the PC component even after injection molding. By local irradiation with a CO_2 laser beam, depending on the laser conditions, conductive tracks with dimensions of about 2 mm width 80 to 370 µm depth and line resistances as low as 1.5 $k\Omega \cdot cm^{-1}$ were created on the surface of the non-conductive plates. Increasing the amount of MWCNT filled PC droplets from 17 to 47 wt% resulted in lower line resistances in the tracks. Comparing two laser parameter sets, lower resistance values of the laser lines were found for a combination of lower laser speed with lower laser power. Higher line resistances were found when the laser lines were created parallel as compared to perpendicular to the direction of the melt flow during injection molding. After irradiation an enrichment of MWCNTs in the laser lines was

detected indicating that conductive paths were generated by percolation of nanotubes selectively within these lines in otherwise non-conductive plates.

Introduction

The efforts to combine the classical setup of electronic circuits with 3 D-components has led in recent years to the development of different procedures for MID applications such as two-component injection molding, hot embossing, film insert molding, mask lighting, the Flamecon-procedure as well as the laser-MID-procedure.[1-8] Thereby, MID stands for "Molded Interconnect Devices" and describes electronic components where metallic conductive tracks are applied to prefab injection-molded polymeric parts. The conductive tracks are created either in a subtractive procedure by removing a previously fully metallized surface or in an additive procedure by means of a selective surface treatment which creates the track structure. A very efficient procedure is the Laser Direct Structuring (LDS) procedure where the laser structuring is carried out on an injection-molded polymeric surface to which an additive has been applied. [9] The activated surface areas afterwards can be chemically copperized, followed by an electrolytically applied layer and finally a 3rd layer of precious metal. However, in the classical MID-technology 4-5 extensive process steps are necessary in order to generate conductive tracks. Investigations on the generation of conductive areas in non-conductive polymers filled with metals and/or conductive polymers have been reported in literature. [10-14]

The utilization of the conductivity of carbon nanotubes was shown for high density polyethylene (HDPE) composites filled with 1-5 wt.% MWCNTs having diameters of 30-90 nm in which the CO_2 laser beam used at constant conditions created conductive v-shaped channels. [15] However, in this study the whole HDPE was filled with MWCNTs requiring relatively large amounts. In addition, a relatively high laser power was applied and relatively deep tracks were generated. By that, undesired conductive behavior within the whole composite material or between the different generated lines cannot be excluded.

To overcome these disadvantages, our study is based on a new concept using nonconductive multicomponent polymer blends with matrix-disperse particle structures in which the conductive nanotubes are located only in the dispersed particles which act as depots. During the laser treatment within the irradiated lines a space resolved conductive path can be created. The local laser activation is expected to melt the depot particles and to move the MWCNTs in the molten lines to the part surface so that a conductive area - a conductive track - is generated by retaining the non-conductivity of the part.

As an example, the blend system of polycarbonate (PC) and polypropylene (PP) was selected. Based on numerous literature reports it is known that amorphous PC is a suitable host for nanotubes and enables relatively good dispersion resulting in low electrical percolation thresholds. [16-20] Therefore, PC was prefilled with industrial available MWCNTs. Polypropylene is a polymer widely used in injection molding applications and was used as polymer matrix. Both polymers are immiscible and form multiphase blend structures. Blends with different compositions of PP matrix and PC-MWCNT particles were used. A total amount of MWCNTs of 3 wt.% was selected, resulting at different PC amounts in different MWCNT concentrations within that component. The main focus of this study is the influence of the PC content and the laser parameters of the used CO_2 laser system on the electrical resistance, morphology and the geometry of the tracks created on the surface of injection-molded plates.

Experimental

Materials and Processing

PP Bormod HF955MO having a density of 0.908 g/cm^{3}was obtained from Borealis Group (Austria). As PC the type Makrolon® 2205 (Bayer MaterialScience, Germany) with a density of 1.2 g/cm^3 and a melt flow rate (MFR @ 300°C, 1.2 kg) of 7 g/10 min was used. The PC filled with MWCNTs was diluted from an industrial available masterbatch Plasticyl™ PC1501 containing 15 wt.% Nanocyl™ NC 7000 provided by Nanocyl S.A. (Sambreville, Belgium) using the named PC type. According to the provider this masterbatch has a melt flow rate (MFR @ 300°C, 20 kg) < 5.6 g/10min [17]. The MWCNTs used for the masterbatch have a purity of >90% [17] and an average length of 1.3 µm [18]. The variation of the compositions compromised different proportions of PP and PC, whereby the MWCNT content was set constant to be 3 wt.% (see Table 1). PC and PC containing composites and blends were dried at 80°C for 2h in vacuum before each processing step.

The blend compounding was done using a Berstorff ZE25 extruder at a throughput of 30 kg/h, a rotation speed of 250 rpm and a temperature profile ranging between 230°C and 260°C using a screw profile typical for the extrusion of polypropylene. Masterbatch granules were fed together with polypropylene and polycarbonate granules into the hopper of the extruder and the extruded strands were cooled in a water bath before granulating. The injection molding to plates with the dimension of 60 x 60 x 2.5 mm^3 was done using a Demag IntElect 80/370-150 with a closing force of 800 kN using a film gate. The conditions were 30 mm/s injection velocity and a melt temperature of 230°C, the tool temperature was 40°C.

Laser treatment

A 30 W CO_2-laser system (Keyence ML-Z9520W) with a wavelength of 10.6 µm in combination with a 3D-scanning unit was used for the laser activation. In summary 11 line structures with a length of 50 mm and a line distance of 5 mm were created on the injection-molded plates (Figure 1). The lines were arranged either perpendicular or parallel to the injection molding direction (melt flow direction). The first line was positioned 3 mm away from the injection gate in the perpendicular arrangement (Fig. 1a) whereas in the parallel arrangement it was positioned 3 mm from the left side of the plate (Fig. 1b). Two parameter sets (PS) with different laser power and scanning speed were used for the laser structuring, whereby the same energy input per length was used. PS 1 involved a medium power (45 %) and a scanning speed of 10 mm·s^{-1} and PS 2 a power reduced to 10 % and a minimized scanning speed of 2 mm·s^{-1}. The laser beam was defocused and had a diameter of 1.6 mm.

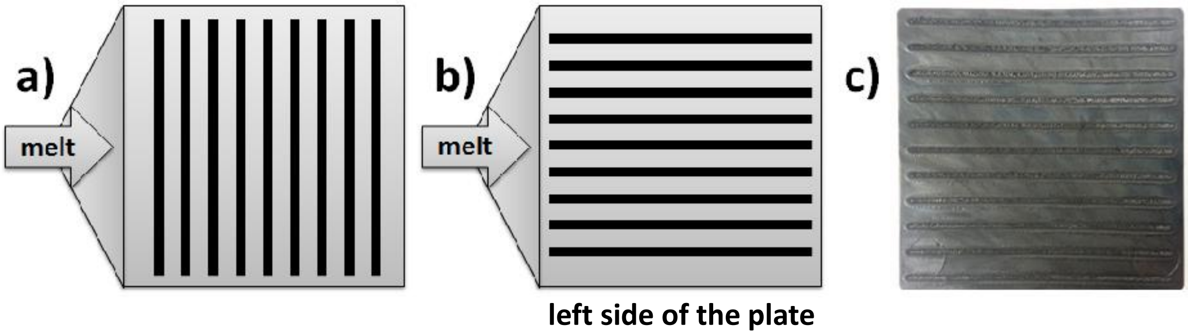

Figure 1. a) Schema for the arrangement of the laser lines when irradiated (a) perpendicular to melt flow direction, b) in melt flow direction with the injection gate on then left hand side and c) photograph of a real sample (60x60mm^2, thickness 2.5 mm).

2.3. Characterization techniques

For optical microscopy (OM) an Olympus BH-2 microscope was used whereby thin sections with a thickness of 5 µm thickness cut at T = -30°C using a Leica RM2265 microtome were observed in transmission mode. Scanning electron microscopy (SEM) was done using Zeiss Neon40 EsB. Cut plain surfaces of the granules were purged for 15 min in chloroform under ultra-sonication to remove the polycarbonate component selectively. Transmissions electron microscopy (TEM) was performed using Zeiss Libra200. Ultra-thin sections with a chosen thickness of approximately 70 nm were produced by cutting at T = -30°C with a Leica UC7 ultra-microtome.

After the laser treatment the injection molded plates showed line-shaped structures with a porous loosely overlying layer which came off by itself or could be removed just by touching. Its occurrence depends on the laser parameters and the material composition. Thus a mechanical cleansing was necessary for a reproducible definition of the geometry and the resistance of the lines which was performed by wiping a tissue over the surface till no black remaining parts were remaining on the tissue.

The electrical volume resistivity of the untreated injection molded samples was determined using a Keithley Electrometer 6517A connected to a Keithley 8009 Resistivity test fixture with ring electrodes (both Keithley Instr. Inc, Cleveland, OH, USA).

The resistance in the lines created by the laser treatment was measured with the 4-point-method using a self-constructed measuring device. By that the track resistance could be determined in Ohm per length unit with an arrangement of six electrodes (Figure 2) combined with a Keithley SourceMeter 2400 (measuring range up to 100 MΩ). Two of the electrodes ($K_{1,6}$) served as a constant power source. The voltage was measured via the remaining four electrodes ($K_{2\text{-}5}$), having an overall distance of 22.8 mm, so that the resistance of the contacted line segments could be defined.

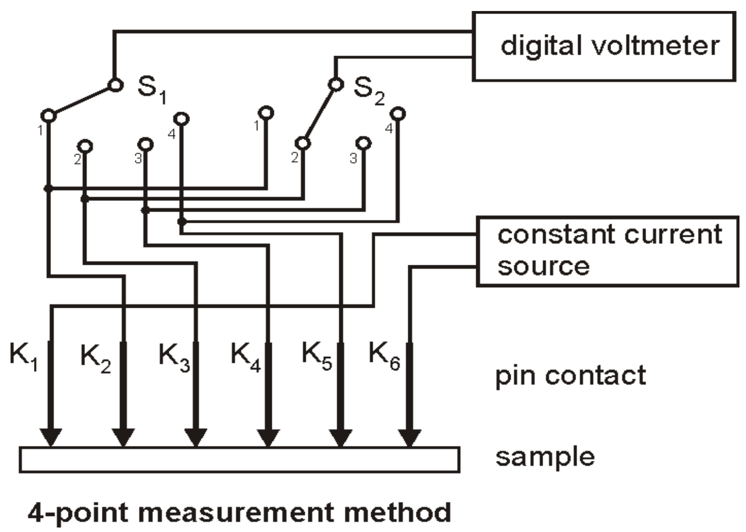

Figure 2: Resistance measurement setup.

For the characterization of the track geometry and the definition of depth and width of the laser activated lines the light microscope Keyence VHX-2000 was used. 3D-imaging of the created tracks was done by using TRACEiT® by Innowep GmbH, Germany. The track roughness was calculated by using Gwyddion Software (Version 2.31). For the calculation only the areas in the created track were taken into account, without considering the bordering track walls.

Results and Discussion

Characterization of untreated materials

The results of the electrical resistivity measurements show that all untreated samples are – as desired – non-conductive (Table 1). With increasing PC content the electrical volume resistivity decreases slightly, but the samples remain to be non-conductive.

Table 1: Samples compositions and their volume resistivity

Sample	PP [wt.%]	PC [wt.%]	MWCNT [wt.%]	MWCNT concentration in PC [wt.%]	Volume Resistivity [Ω cm]
PC17	80	17	3	15	9.1 E14
PC22	75	22	3	12	1.3 E15
PC27	70	27	3	10	6.2 E12
PC37	60	37	3	7.5	2.5 E11
PC47	50	47	3	6	9.49 E11

Observation with optical microscopy of untreated samples (Figure 3, left) shows that the blend morphology is determined by a homogeneously distributed dark appearing phase. With TEM it can be seen (Figure 3, right) that the dark appearing phases represent PC particles in which the MWCNTs are localized selectively, which explains the insulating behavior of the injection molded samples. After removing the PC-MWCNT component with chloroform and investigating the remaining parts with SEM (Figure 3, middle), fine dispersed particles with diameters up to 30 µm (sample PC22) can be observed. With increasing PC content, more numerous PC particles with larger sizes and smaller distances between them can be observed, however still separated by insulating PP matrix (not shown here). At the same time the filling level within the disperse PC component decreases, but being still above the electrical percolation concentration as reported in literature for PC. [16-20]

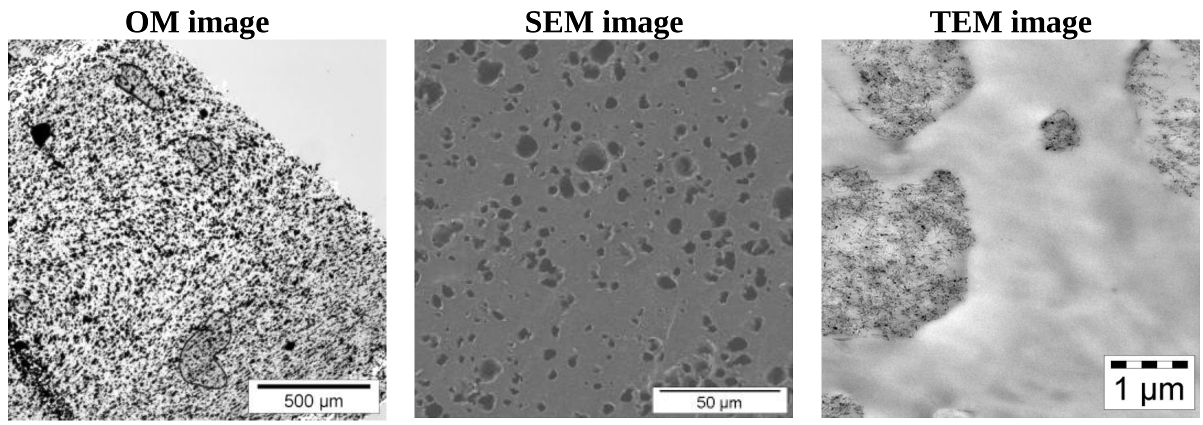

Figure 3. Microscopy images of PC22: transmission optical micrograph of a thin section (left), SEM image of a cut surface with removed PC component (middle) and TEM image of an ultrathin section (right).

Resistance within the laser lines

A laser treatment with the above-mentioned parameters led in all cases to electrical conductivity of the activated areas. The resistance within the laser lines depends on the laser parameter and the material composition as well as the positioning and arrangement of the lines on the injection-molded plate. Figure 4 shows the determined resistance values of the lines created with different laser parameters and arranged perpendicular to the melt flow direction. For both parameter sets the line resistance ranges from 1.5 to 40 $k\Omega \cdot cm^{-1}$. It drops from sample PC17 to PC47, even if the MWCNT concentration decreases significantly in the

PC part (see Table 1). However, obviously the denser packed larger dispersed particles at higher PC contents enable a more homogeneous formation of the conductive tracks so that the line resistances are lower in those cases. The lines created with PS 2 at lower laser power and scanning speed generally show lower resistance values compared to PS 1. For blends with a PC content ≥ 27 wt.% the resistance values vary between 1.5 and 10 kΩ·cm^{-1} for PS 1 and between 1.5 and 2 kΩ·cm^{-1} for PS 2. The influence of the laser parameters on the achievable resistance seems to be lower for materials PC37 and PC47 having higher PC contents than for the materials PC22 and PC27. There is not much difference between PC37 and PC47, whereas for the samples PC22 and PC27 with lower PC content a considerable reduction in resistances from PS 1 to PS 2 was found. For sample PC17 with the lowest PC content and the largest distance between the dispersed filled PC particles a clear dependence of the resistance on the line position could be observed. Resistance declines with the distance to the injection gate which indicates an inhomogeneous distribution of the PC-MWCNT particles in the injection-molded, and possibly also differences in size and elongation of the dispersed filled PC particles in dependence on the flow lines [19] within the injection molded plate.

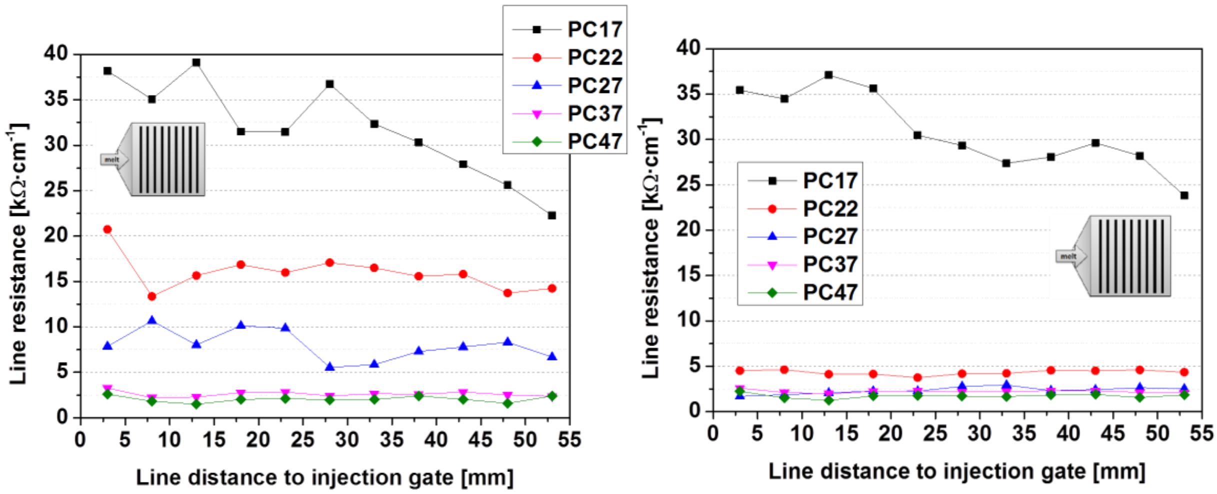

Figure 4. Resistance of lines as function of line position perpendicular to flow direction at different blend composition: parameter set 1 (left), parameter set 2 (right).

The results of laser structuring in the direction parallel to the flow of the material during the injection molding process are show in Figure 5. For the blends with a PC content ≤ 22 wt.% (samples PC17, PC22) significantly higher line resistance values (15 - 160 kΩ·cm^{-1}) with considerable variations were measured compared to those where lines are arranged perpendicular to the melt flow direction. When the PC content is higher, the activated lines show similar resistance values (1.5 – 10 kΩ·cm^{-1}) for both line arrangements. Also when the lines are parallel to the flow direction, resistance decreases with an increasing proportion of PC-MWCNT component again indicating higher homogeneity and easier path formation when larger MWCNT filled particles are closer together before irradiation. For PS 2 generally a lower resistance of the lines than for PS 1 was found. An exception is material PC22, where the values for PS 2 are above those of PS 1 and sometimes even exceed the measuring range (100 MΩ). For blends with a PC content ≥ 27 wt.% the resistance values vary only slightly between PS 1 (2 – 8 kΩ·cm^{-1}) and PS 2 (1.5 – 6 kΩ·cm^{-1}).

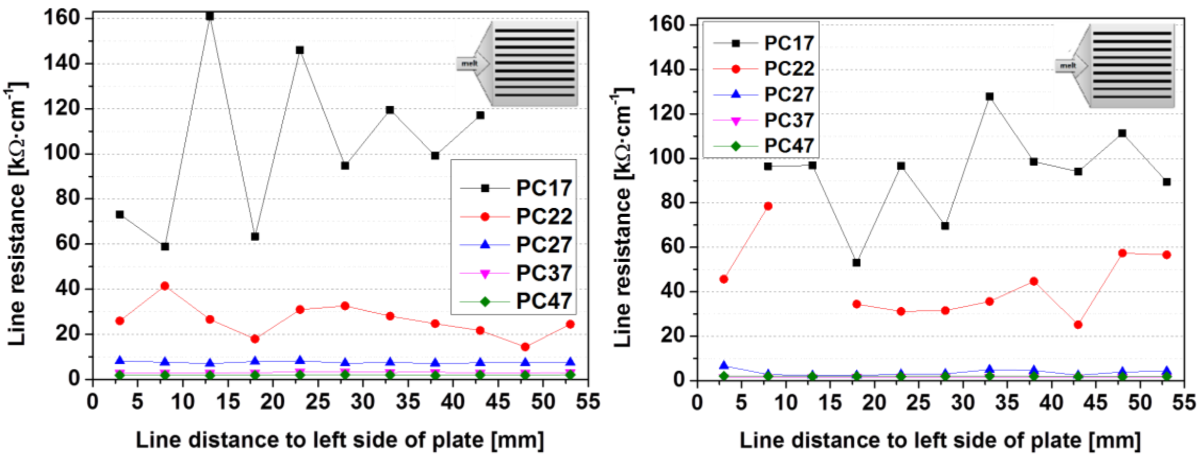

Figure 5. Resistance of lines as function of line position in flow direction and blend composition: parameter set 1 (left), parameter set 2 (right), for arrangement see Figure 1.

As the test results show, the line resistance decreases at both line arrangements and parameter sets with increasing content of the PC-MWCNT component. To evaluate the influence of the PC content on the obtained line resistances, the measured values at 33 mm distance to the injection gate (Figure 6, left) and 33 mm to left side of the plate (Figure 6, right) were compared. For both laser directions and parameter sets a decrease of the line resistance is obtained with increasing PC content whereas the lines were generated perpendicular to the melt flow direction exhibit slightly lower values when generated with parameter set 2.

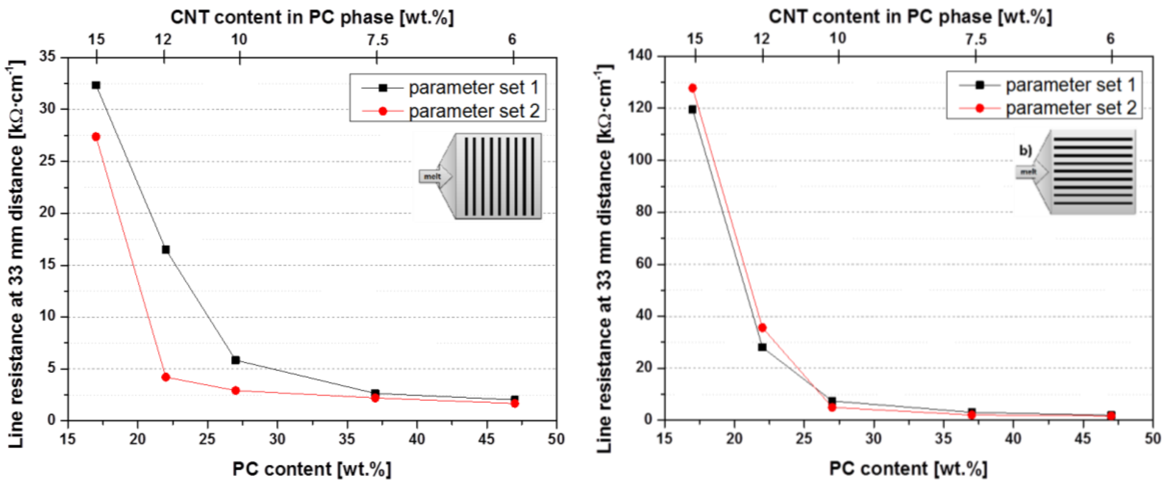

Figure 6. Line resistances at position 33 mm as function of PC content: perpendicular to melt flow direction (left) and in melt flow direction (right).

As the distance between the MWCNT filled PC particles is lower at higher PC loading, the creation of a conductive structure through laser treatment seems to be easier and more homogeneous tracks with lower resistances are achieved.

For blends with PC contents ≥ 37 wt.% the resistance values for PS 1 get more and more similar to those of PS 2. One may this assume that the electrical conduction effects take place not only in areas close to the surface but also in deeper areas of the material.

The differences and variations of the resistance values regarding the line arrangements for blends with lower PC contents can be traced back to an inhomogeneous distribution of the PC-MWCNT particles in the plates resulting from the injection molding process, which was hold constant in the study.

Geometrical and morphological analysis of the conductive lines

The tracks have a v-shaped geometry with different steep slopes and material bulging on the edges (Figure 7). Due to the injection molding process a stretching of PC-MWCNT particles in melt flow direction occurs (Figure 7, left) which results in oblong filled structures. This effect is more pronounced at the samples edges due to the fast cooling process of the material at the cavity. The stretching of PC-MWCNT particles vanishes in the samples core, where the material during the cooling process found time for a shape relaxation of the deformed drops.

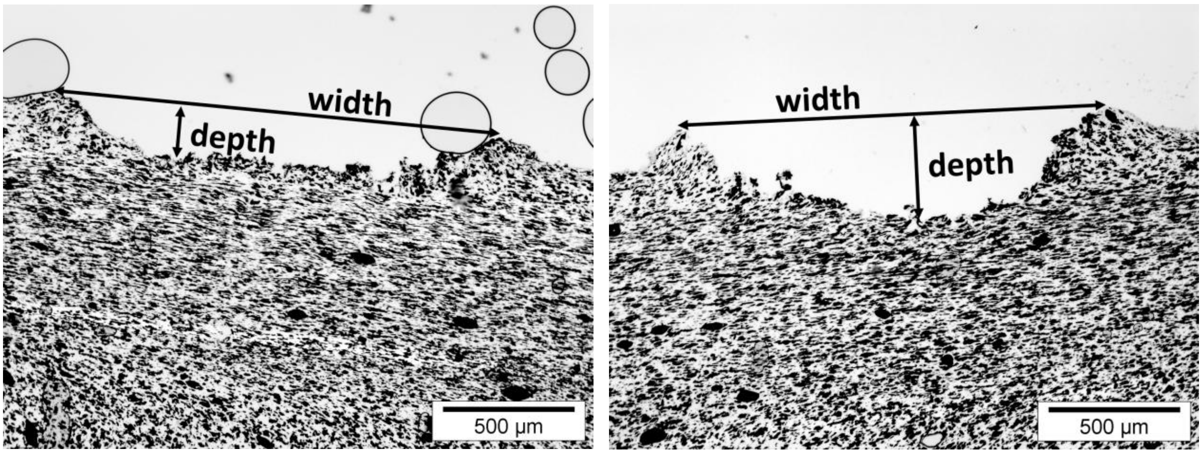

Figure 7. Optical micrographs of cross sections of created lines for PC27 blend system in melt flow direction: comparison of laser parameters PS 1(left) and PS 2 (right).

TEM investigations on the generated tracks show an accumulation of MWCNTs directly at the surface in the created laser lines, representatively shown for PC27 (Figure 8). These accumulated MWCNTs enable the electrical conductive behavior in the generated tracks.

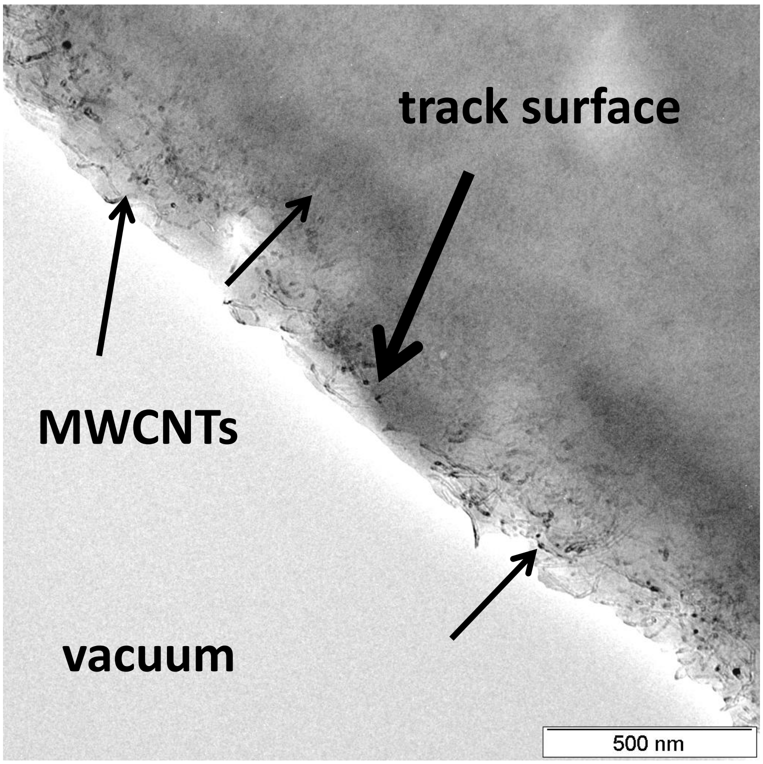

Figure 8. TEM image of accumulated CNTs at the track surface in PC27 blend system, generated in melt flow direction, some nanotubes are assigned by arrows.

The line depth and width vary depending on the laser parameters and the blend composition (Figure 9). Line depths of 80 µm to 230 µm were determined for parameter set 1. Reduced laser power and scanning speed (PS 2) lead to larger line depths ranging from 180 µm to 370 µm. In both cases the line depth diminishes from sample PC17 to PC47, i.e. with an increasing content of the PC-MWCNT component. The line widths range for PS 1 from 2070 µm to 2350 µm and for PS 2 from 1750 µm to 2000 µm. A dependence of the line width on the material composition could not be found.

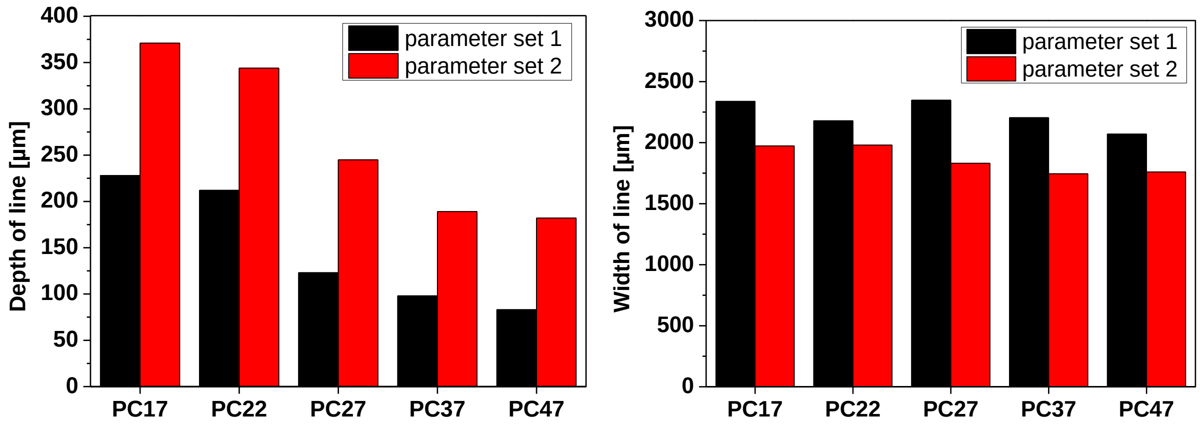

Figure 9. Geometric dimensions depth and width of the created conductive tracks at different blend compositions.

Due to the reduced scanning speed when using PS 2 more energy reaches deeper areas in the material than using PS 1. This leads to a higher line depth and thus also a larger activated area of the v-profile. Assuming that a conductive structure is created on or close to the surface a larger activated area results in higher conductivity and thus lower resistance.

Due to the same energy input per length in the samples for PS 1 and PS 2 it can be assumed that the different line depths and widths depending on the laser parameters are due to the different heat conductivity and thermal stability in the interaction zone of laser radiation with the polymer blend. While for PS 1 the radiation is absorbed by the material within a short period of time and the thermal energy is transmitted only in areas close to the surface, for PS2 the material is exposed to the laser radiation 10 times longer and the thermal energy is transmitted into deeper areas of the material. The reduction of the line depth with an increasing content of PC-MWCNT component is a result of the higher melting and decomposition temperature of PC compared to PP, which leads to a lower proportion of melting material with the same energy input but a higher PC content. Beside the differences in line geometry, the two different laser parameter sets cause a different roughness in the conductive lines. In Figure 10 a 3D-image of a generated track created perpendicular to the melt flow direction in the sample PC17 is shown. The calculation of the area between the bordering steep walls resulted in a roughness of Ra = 27 µm generated with parameter set 1. In the case that the track was generated with the parameter set 2 a roughness of Ra = 43 µm was measured.

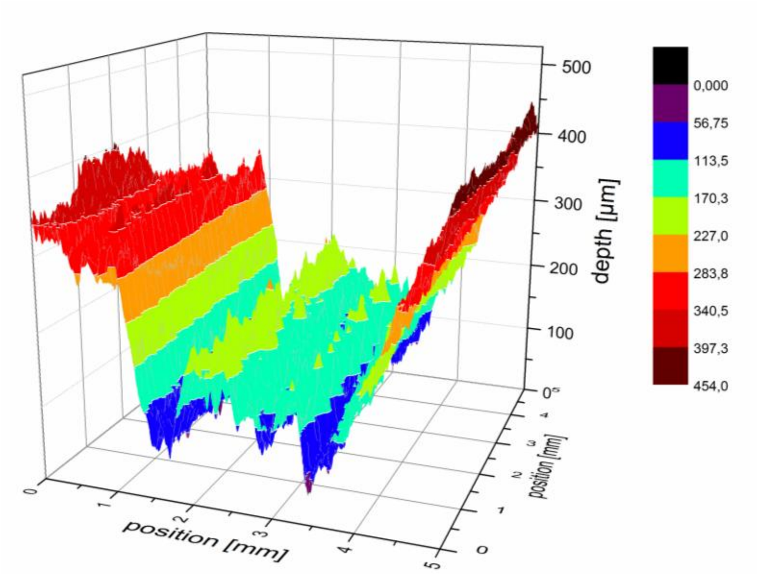

Figure 10. 3D-image of conductive track in PC17, generated with parameter set 1 perpendicular to melt flow direction.

Conclusion

Electrically insulating polymer blends, consisting of a polypropylene matrix and dispersed polycarbonate particles filled with 3 wt.% multiwalled carbon nanotubes, were locally activated through IR-laser radiation. This created electrically conductive line-shaped structures on the surface of injection molded plates. Morphological investigations of the injection molded samples revealed a selective localization of the MWCNTs in the PC component. An accumulation of MWCNTs in the laser lines could be detected, which enabled a space-resolved conductivity within the activated areas. Resistivity values depend on the blend composition, the laser parameters, and the arrangement of the lines on the plate which was either parallel or perpendicular to the melt flow direction. Increasing PC content of the polymer blends reduces the resistance of the laser-activated lines, although the MWCNT concentration in the PC component decreased. This can be explained by the blend morphology, having larger and more closely packed MWCNT filled PC particles at higher PC loadings, which however are still separated by the insulating PP matrix. With such a starting blend structure it seems to be easier to created homogeneous conductive tracks. A combination of low laser power and low scanning speed leads to a low resistance values. In laser lines generated perpendicular to the melt flow direction lower resistance vales were found in comparison to laser lines in melt flow direction.

The results imply that even lower contents of MWCNT could be enough to create conductive laser lines. Further examinations regarding the injection molding parameters are planned to deeper clarify how the differences between different PC amounts in the blends are dependent on the blend morphology. In view on the planned injection molding of more complex part geometries enabling structuring with laser treatment also in 3D, homogeneity of the blend morphology can be important, which possibly requires the use of blend compatibilizers. In addition, laser treatment conditions should be optimized in order to get smaller conductive tracks and lines with higher conductivity.

In summary, the study shows that conductive, metal-free tracks can be generated by localized laser treatment of a non-conductive immiscible polymer blends with an insulating matrix and conductive dispersed particles. This seems to be a promising way in the direction of cheap and easily processable MID applications.

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Table of Content (TOC)

Electrical conductive tracks can be created by local laser beam irradiation of injection molded plates consisting of immiscible two-component polymer blend in which the proportion between polycarbonate and polypropylene is varied. The nanotube localises in PC and after laser treatment an accumulation of MWCNTs directly at the surface in the created laser lines can be observed.

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ToC figure