THE ADDED VALUE OF 3D POLYMER DEPOSITION ON TEXTILES

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Abstract: *The working hypothesis for this research project is that it is possible to develop a new functional polymer printing process for the direct application of conductive polymer onto textiles. We will use the basic extrusion technology that is currently applied in 3D printing. Thus the aim is also expanding the knowledge and knowhow base of 3D printing and make this technology applicable for deposition of functional polymers on textiles in such a way that process parameters are clearly understood, and pre-defined final product specifications can be met. Thus the challenge is to apply conductive tracks with a simple one step process that fits the current textile production processes. This means that investigating polymer deposition onto textiles of bio based polymers like PLA, doped with carbon could be a versatile route to achieving economic and sustainable conducting textiles. If the mechanism underlying the bonding of doped PLA with textiles can be controlled for processing then a new route to achieving conductive grids would be opened.*
Keywords: *3D printing, modelling polymer deposition, functional polymers, conducting textile grid, added value,*

1. Introduction

The textile industry has is a continuous demand for more efficient, environmental friendly production processes. Also there is a continuous search for innovative functional properties that have added value for the end users. New (bio based-) materials, conductive polymers, shape memory polymers and the like promise advanced application opportunities for innovative textile companies. It is expected however that these materials put an even heavier demand on the industry for specific functionalities that can only be incorporated in textiles if in depth knowledge of processing and application of functional materials is available [1]. This is relevant for smart- or intelligent textiles where conducting fibres or yarns are incorporated into the fabric or attached to the surface. In addition electronic components are fitted to the fabric to either act as sensor devices or as actuators. Next to this, for control, processing units are used, often mounted onto a mini printed circuit board. Altogether in these applications we are dealing with textiles that are modified in such a way that flexibility or drape may be influenced in a negative way. Depositing functional polymers may thus influence the drape of a fabric in a negative way and consequently we need to know how critical this may be and this is one of the subjects in this research project.
To meet the needs for flexible conductive grids or arrays combined with textiles in order to develop the smart or intelligent textile markets represents an important area of research. There are a number of prominent ways to achieve these goals and the following three strategies seem dominant:

- Direct printing of conductive particles on textiles
- Functional conductive polymers
- Integration of metal conductors with textiles

For this research we focus on functional conductive polymers and there are many electro-active polymers currently being developed by the electrochemistry industry, a technology convergence of electronics and chemistry/polymer sciences, for what is called plastic electronics.

Printing or deposition of conductive arrays on textiles is challenging since textiles are flexible 3D structures with voids and fibres that hinder interconnection of the conducting particles of the ink.
An example of printing of conducting array on untreated rigid surfaces is the research by Van Osch et al: Small conductive tracks are created by direct inkjet-printing of an ink with 30 nm silver particles onto flexible and transparent untreated poly(acrylate) foils with a low surface energy. Lines with a diameter as narrow as 40 micrometres are obtained. After sintering, the conductivity of the obtained silver tracks is 13 to 23 % that of bulk silver. Such direct inkjet-printing may be applied in plastic electronics, where pre-structuring or pre-treatment of the substrate should be avoided to reduce production costs [21,22]. However this requires a stable non flexible substrate and the method cannot be extended to textiles.
Printing of carbon nanotubes and silver nanoparticles on textiles has been studied. These inks are commercially available from: Bayer Material Science, Aldrich, Applied Nanotech Holdings, and Suntronic. Conductivities are in the order of 10^3 S/cm for silver nanoparticles to even higher values for CNTs. Unfortunately, when the conductive track was printed on textile substrate, the conductivity was lost owing to several factors like surface discontinuity because of the 3D and flexible nature of the textile substrates, substrate absorbency and the large distance between the ink drops [23]. Only very thick layers covering the whole of the surface, proved to work (unpublished).

CONNECTOR TECHNOLOGY: Connectors are required when a conductor needs to connect to different electric devices or a different part of the circuit. Improper interconnection causes incomplete contact and non-uniform resistance at the connecting point. Below an account of a few tests [24].
Welding provided a reliable electrical connection, good strength at the junction and good electrical conductivity. However at the junctions a blob of material was formed which provided a bending point. After repetitive bending the wire would break. This method is also applicable for the connection of polymeric conductors based on thermoplastics. Above their Tm the molten parts are presses together and after cooling down bonding is achieved. However, this can only be successful if chemically similar polymers are being used.
Stapling or clamping is very useful in terms of increasing flexibility at the junction points. However the rigidity of the staples may accelerate fabric tearing. This means there is a possibility of the stitches coming loose. This resulted in an unstable connection.
Conductive yarns can also be connected by means of conductive adhesives. Conductive adhesives allow the conduction of electricity, making it ideal for connecting conductive yarns. They are manufactured using micro carbon technology. Conductive adhesives can be envisioned that are nontoxic, highly conductive, highly durable, and moderately flexible. This is also a good option for joining polymeric conductors.

MODEL DEVELOPMENT: In this part we only discuss the overall modelling of the system. Details and mathematical background will be elaborated in later research reports. However, bonding experiments and penetration of the model polymer will be subjected to modelling starting from the earlier developed models as discussed below.
Sharpe and Schonhorn developed the adsorption theory where a number of bonding forces are considered [32]. In this model van der Waals forces and hydrogen bonds are included. The adhesion process was described by the Dupre equation:
Work of adhesion : $Wa = \gamma p + \gamma s - \gamma ps,$ eq. 1
where γ_p and γ_s are the surface energies of the polymer and substrate respectively in contact, and γ_{ps} is the interface energy. Combining this with Young's equation we can write the work of adhesion as
$$Wa = \gamma p(1 + cos\theta) \qquad \text{eq. 2}$$
with θ being the contact angle between polymer and substrate. However there are examples of systems where spreading of polymer over a substrate is complete (PE on glasfibre) but adhesion is very low and, reverse, where spreading is minimal but adhesion is high (Polysulfon on aramide) [33].

It can be shown [10],that in practise the combination of eq.1 and eq. 2 reduces to:
$$Wa = \gamma p + \gamma s - \gamma ps \approx 2[(\gamma p^d \gamma s^d)^{\frac{1}{2}} \qquad \text{eq. 3}$$
Where the superscript d refers to dispersion forces, γp and γs can be found in the literature, e.g. in Van Krevelen, p 240 [10] or measured using inverse gas chromatography [34]. By measuring bonding forces and taking into account the physical characteristics of our test materials we will be able to predict bonding characteristics of our model systems.
However, bonding is only one part of the total complex of properties we are dealing with. We also have to consider transport of the melt into the textile substrate. For this we consider the pores in textiles as capillaries.
The Lucas-Washburn equation [35], also known simply as Washburn's equation, describes the rate dh/dt of fluid flow through a cylindrical capillary of radius r as a function of the driving pressure. Making the assumption that flow is laminar viscous and incompressible, Washburn applies Poiseuille's Law for the pressure drop in a fluid flowing through a cylinder to derive the following equation:
$$\frac{dh}{dt} = \frac{\Sigma P}{8\eta h}(r^2 + 4\lambda r) \qquad \text{eq. 4}$$
where η is viscosity and ΣP is the sum of atmospheric pressure (zero if the ends of the capillary are open), hydrostatic pressure, and capillary pressure, λ is the coefficient of slip, taken to be zero for a fully wettable surface. Capillary pressure Pc is:
$$Pc = \frac{2\gamma \cos\theta}{r} \qquad \text{eq. 5}$$
where γ is surface tension and θ is the solid-fluid contact angle [35,36]. In our system we have to add an additional pressure. Thus ∑P = Pc+Pu, where Pu is the external pressure applied.
Thus our starting equation becomes:
$$h^2 = \frac{(\gamma.cos\theta) + Pu}{4\eta}(r + 4\lambda)t \qquad \text{eq. 6}$$

In this research polymer melt must be able to penetrate the fabric in order to form a fixed adhered bonding package between the molten polymer and the fabric after solidification of the polymer. Gooijer analysed flow resistance of textiles [37]. Since in this research flow through the fabric is not the objective but flow into the fabric is, the pore models seems more appropriate for our goal.

Important in the analysis of Gooijer is the permeability k, that can be expressed as:

$$k = \frac{\epsilon dp^2}{16k0} \qquad \text{eq. 7}$$

where ϵ is the porosity of the fabric, dp is the pore diameter and k0 is a correction factor, the Kozény constant. This expression for k is valid for porosities smaller than 0,8 which for most wovens is the case [37]. However in our test systems we are dealing with relative high viscosities thus we have to apply additional pressure to achieve better penetration. Thus the subject of further research is also to adapt the above modelling and make it fit our systems.

2. Creating added value

Smart functional materials represents a huge area of research and development. Literally thousands of prototypes have been developed, but market introduction on a large scale of products based on this technology has not happened yet. To quote the Systex vision paper: “The key market hurdles for development and subsequent commercialization of smart textiles applications are of technical, strategic and economic nature: The technological barriers include reliability, durability and ease of use; slow development in the areas like flexibility of electronics, durability and power; and missing interconnection of components. The business barriers include high development and manufacturing costs; high retail prices leading to less consumer acceptance, scarcity of human resources to carry out new product development” [2].
Thus although the amount of progress in smart textiles is remarkable in terms of demonstrators, many of these developments are not suited for large scale production [3]. When a conductive grid is incorporated or integrated with textiles only part of the problem is solved, it requires the ability to connect these conductors with each other or with electronic components, and energy supply like batteries on (semi-) automated production scale. Wearable electronics have to be worn by persons without compromising on quality, comfort and fit (drape is important). This problem is as yet unresolved and is one of the key issues that hinders large scale introduction. There is a need for the development of new high-tech materials with special properties in combination with new processes delivering functional additives maintaining characteristics of the textile material. The current state of technology requires that textile is pre-treated to achieve sufficient printing results caused by typical textile properties and texture. In addition, fabric pre-treatment is necessary [4].
3D polymer printing, or better, polymer deposition, is a relatively new technology that enables the deposition of functional polymers on a surface in a programmed way in three dimensions. It is used abundantly in areas like rapid prototyping [e.g.5]. Usually the material is printed onto a smooth surface that is not part of the final product and that acts as a temporary carrier only. There are only a few random examples of 3D polymer deposition using print technology on textiles: as yet an unexplored field [6].
These 3D structures may find potential application as conductors and sensors and may lead to the possibility of adding functional properties to substrates, in our case textiles. Several strategies have recently emerged for precisely assembling three-dimensional periodic arrays, including direct-write techniques (a form of 3D deposition). This approach offers the materials flexibility, low cost, and ability to construct arbitrary three-dimensional structures required for advances across multidisciplinary boundaries [6,7]. Several direct-write techniques have been introduced that are capable of patterning materials in three dimensions.

3D application of functional properties on textiles is not trivial due to the manifold of interactions and binding/adhesion phenomena that occur at the various interfaces. The fact that textiles are flexible, composed of a wide variety of different polymeric materials, prone to several constructions and cloth architectures, and composed of yarn, fibres and filaments makes proper understanding of the interface phenomena a real challenge. Until the processes that play a dominant role in these interfaces are really understood, processing on an industrial scale is limited to trial and error leading to non-generic, ad hoc solutions that cannot be generalized to standardized production processes. These are required for larger scale industrial processes. This requires the development of conductive polymers that can be deposited directly on the textile though simple extrusion directly on the textiles, if possible without additional pre-treatments or smoothing coating layers [8,9,10,11]. Most commercially available polymers are non-conducting insulators or only marginally conductive.
In the iPolycond project [12] polymers doped with carbon nanotubes have been studied for application in conductive composites with promising results. This means that investigating polymer deposition onto textiles of biobased polymers like PLA, doped with carbon could be a versatile route to achieving economic and sustainable conducting textiles. If the mechanism underlying the bonding of doped PLA with textiles can be controlled for processing then a new route to achieving conductive grids could be opened.

3. Materials and Methods

We investigate the use of systems that apply polymers through dosing or metering devices, such as digitally controlled printheads (Some preliminary results are show in fig.1). This concept was already proposed in the early 1990-ies [7], but has developed at high speed leading to the implementation of ‘direct-write’ rapid prototyping technology that we nowadays see used globally [7]. Alternatively hot melt printing has been conducted by various groups [13,14,15,18]. At present we see a rapidly developing trend in the area of 3D printing techniques that allow for low cost manufacturing of objects [6].

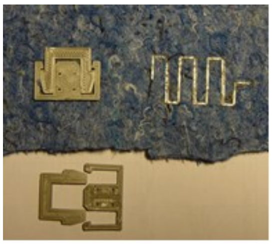


Fig. 1. First results

Polymer is fed into an extruder-heating system for deposition of low melting temperature thermoplastic e.g. PLA on a surface [16]. It should be noted here that all research and developments in this field are aimed at producing isolated products without any supporting substrate. In fact the non-interaction with the supporting substrate for these applications is considered as a positive aspect [17]. For the model system employed in our research an existing 3D polymer deposition system will be used that we want to adhere to the substrate. In the envisaged experimental and production set up a polymer molten by an extruder head is deposited onto the textile substrate (see schematically fig. 2 [19]). Intrusion into the fabric is required for fixation or anchoring of the polymer, since that will provide for a larger contact area, the interface, and thus the total bonding energy will be enhanced by spreading of the polymer over the accessible surface. If the results of his research are to be applied by the industry, fabric design will be important and that will mean that the polymer filaments deposited will not be placed at random but at carefully selected positions. It is foreseen that he exact positions will coincide with intended folds and close to stitches. The impact of polymer filaments on drape and bending will be an important evaluation criterion.

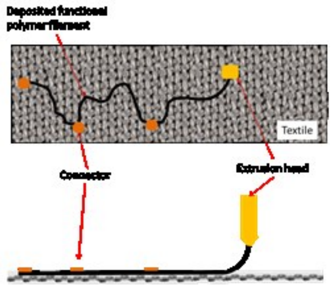
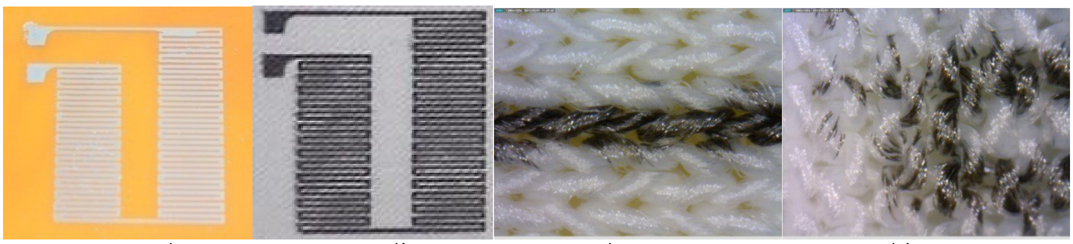


Fig 2. Schematic set up

4. Results and discussion

As first results of this research we tested printing of conductive tracks on textiles [25]. A typical example of the issues encountered when printing on textiles is presented in fig 3 a-d. The prints in Fig 3a and 3b were references printed on plastic sheets. Carbon particles dispersed in an inkjet fluid, resulting in conductive ink, was printed of different substrates. The test was temperature increase at low voltages. On a polymeric substrate (fig.3a) already at a low voltage a high temperature was obtained (4 V implied a 30 ˚C temperature), however a small increase in the intensity of the current of around 10 mA, made the temperature rise with more than 10˚C. This leads to the conclusion that with a small capacity battery, great heating elements can be achieved via conductive inkjet printing.



1a 1b 1c 1d

Fig. 3. Inkjet printed tracks on polymeric films (a), PES-substrate (b) and ink spreading on PES substrate (c,d)

Unfortunately, when the conductive track was printed on textile substrate, the conductivity was lost owing to several factors like surface discontinuity because of the flexibility and the 3D nature of the textile substrates. Therefore a droplet test was performed to investigate the possibility of achieving a conductive track, printed on classic textile substrate that would be not only conductive, but also resistant to stretching. Thus, an ink droplet of around 1 ml was placed on a cotton and PES substrate, with the aim of obtaining full surface coverage. The cotton samples still did not show any conductivity, but the ones printed on PES substrate did. Its resistance ranged 100-200 Ω, even after repeated vertical stretching [25].

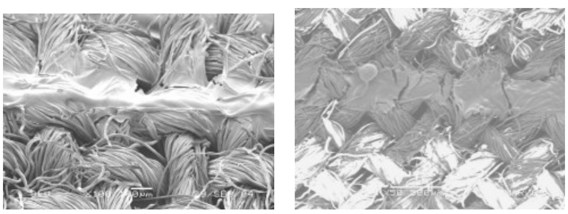


Fig. 4. PEDOT-printed cotton before and after 10% elongation [26]

Research by Amit Sawhney et al [26] on printing of thin layers of a suspension of Poly (3, 4 – ethylenedioxythiophene) - poly (4-styrenesulfonate) (PEDOT-PSS), 1.3 % by weight, onto mercerized cotton fabric for 500 cycles. Fig. 4 shows some of their results. The cracks in the deposited PEDOT where reversible but while stressed the conductivity reduced by 20% or more.

For proper understanding the underlying mechanisms of polymer-textile bonding we conduct the following preliminary experiments:

1. Production of carbon loaded PLA. The polymer used was PLA 4043D general purpose PLA form nature works. The carbon dopes were:

Ketjen carbon black EC 600 JD, surface area 1400 m2/g. Particle size: 68nm. Supplied by Akzonobel, Ketjen carbon black EC 300 JD, surface area 800 m2/g. particle size: 80nm. Supplied by Akzonobel, and carbon fibres, length: 0,1 mm, cross section: 0,007mm. Supplier: R&G Faserverbundwerkstoffe GmbH. For detailed product specs see: [27]. All mixing experiment were carried out on a Berstorff extruder. The extruded doped PLA was chopped into a fine granulate for further processing. The extruder temperature zones were from motor to nozzle set at 30, 120, 170, 200, 210, 220, 200,and 200°C respectively. Concentrations were controlled by controlling the feed mass flows by gravimetric control. The PLA flow was set at 8 and 9 kg/h (depending on the experiment) and the carbon flow at 2 resp. 1 kg/h resulting in concentrations of 20 or respectively 10% of carbon loading. These experiments will be extended to other polymer/carbon systems. The resulting materiel will be re-extruded and two concentration ranges will be produced: 15 and 7,5% for carbon black EC 600 JD and 10 and 5% for Ketjen carbon black EC 300 JD and the carbon fibres. Conductivity measurement before and after deposition textiles will be carried out using the two probe method [28]. To test the performance of the system determine the change in resistance as a function of strain on the textiles expressed as the Gauge factor (GF). This factor is the ratio of relative change in electrical resistance to the mechanical strain ε, which is the relative change in length [30,31].

$$GF = \frac{\Delta R/_R}{\varepsilon} \qquad \text{eq. 8}$$

Where GF is the gauge factor. ΔR/R is relative change in resistance as a result of the strain ε= ΔL/Lo. The effect of stretch will be evaluated since in the combination with textiles that is a critical important parameter. In addition the bonding strength between extruded filament and the substrate (100% Cotton, 50/50% polyester /cotton, and 100% polyester) will be measured. These measurements are subject of further research.

2. In a second series of experiments we investigated the penetration of molten PLA into a bundle of polyester (PET) fibres. These tests must be seen as model experiments to investigate the flow- and bonding characteristics of molten PLA into a complex capillary bundle of fibres. This series of experiments was conducted using Synterra PLLA 1510 (MFI 8±2) and Synterra PLLA 1010 (MFI 22±5) (Synbra). For specs see [29]. Deposition experiments continued with the Brabender lab extruder / Plasticorder,. The extruder was fitted with a vertical extruder head. Spindle speed was set at 75 rpm for Synterra PLLA 1510 and at 60rpm for Synterra PLLA 1010, the extrusion temp was set at 210-220°C for both types of PLA.

PET Bundles studied are specified in the table 1.

Table 1. PET bundle specs

	PET bundle specs	
Average weight per fibre	0.00188 ±0.0005 g (n=300), based on n=300: 0.00188 ± 10%	
Average fibre diameter	0.1792 ± 0.01 mm (n=25) Cross section area per filament 0.025 ± 0,003 mm^2	
Weight of bundle	20 mm bundle: 10.0g ±0,1, n=11	26 mm bundle: 20,15g ±0,04, n=11
Nr of fibres per bundle	20 mm bundle: 5319 ± 1415	26 mm bundle: 10718 ± 579
Surface area bundle	307,75mm^2,	530,66 mm^2
Surface occupied by fibres	133 ± 39 mm^2	268 ± 36 mm^2
Void surface	176 ± 52 mm^2 or 57 ± 17%	263 ± 38 mm^2 or 50 ± 7%
pressure	3,25 kg/cm^2	1,5 kg/cm^2

At first the PLA was allowed to freely flow onto the bundle. But as can be seen from fig. 5a, no penetration or bonding between the molten PLA and the PET bundle took place. The next experiments were performed with pressure on the melt immediately after applying the melt. In this case the pressure ranged from 1,5 to 3,25 kg/cm^2. As can be observed from fig. 5 b-d some penetration and bounding took place. Extensive evaluation is subject of further research.

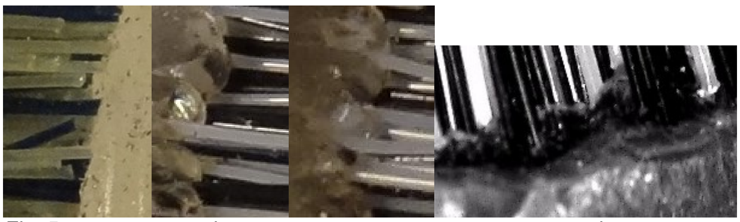


Fig. 5 a b c d
PLA mass in contact with PET fibre bundle

The results of this project will enable the textile industry, the textile supply industry and the whole array of textile related industry to optimise their R&D&E process in the area of 3D printing of functional polymers. The knowledge generated in this project will strengthen the position of the textile finishing industry in an international context. The ability to make recycling possible is an important boundary condition.

5. Conclusion

Polymer deposition on textiles is not trivial, requires specific processes, and depends strongly on the combination textiles-polymer. The area includes research into new polymers/materials, polymer-textile adhesion, and deposition/extrusion technology. Applications and the possibility to incorporate these into textile rely on the fundamental insight in, and subsequent control of, the mechanisms that influence surface phenomena of these textile materials. We foresee that 3D polymer deposition will contribute to the possibility of developing innovative products, like comfortable intelligent workwear with optimal drape. But before these can be produced true understanding of the surface and adhesion phenomena of polymer melts deposited on textiles is necessary.

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