Master's Thesis Engineering Technology

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In Situ Measurement of Percolation Threshold of Conductive Fillers and Integration into a Self-Healing Elastomer

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INTRODUCTION

IMO-IMOMEC is running an investigation to optimise the design in printed structures and read-out electronics for the construction of flexible tactile sensors. Principally, it focusses on the development of stretchable electronic conductors by the deposition of a conductive blend. An elastomeric matrix (polydimethylsiloxane) alters from insulator to conductor by increasing the conductive filler load until the percolation threshold is passed [1]. The first objective is designing an in situ measuring tool to quantify permanently the blend resistance.

Unfortunately, cracks can arise in the conductor while stretching and it results in permanent damage and loss of conductivity. Due to this crack formation, it is necessary to synthesise a conductive matrix with good blending characteristics, a low electrical resistance and reliable elastic properties. The second objective is to check if a self-healing elastomeric matrix, which is able to repair little cracks without external aid, meets these requirements by the integration of conductive fillers.

EXPERIMENTS

PERCOLATION EXPERIMENTS

The in situ measuring tool (Figure 1) consists of 4 tungsten electrodes, which are attached in the bottom of a round bottom flask. Due to this design, a four-wire measurement quantified in situ the blend resistance (as a function of the volume percentage of conductive fillers) to determine the percolation threshold.

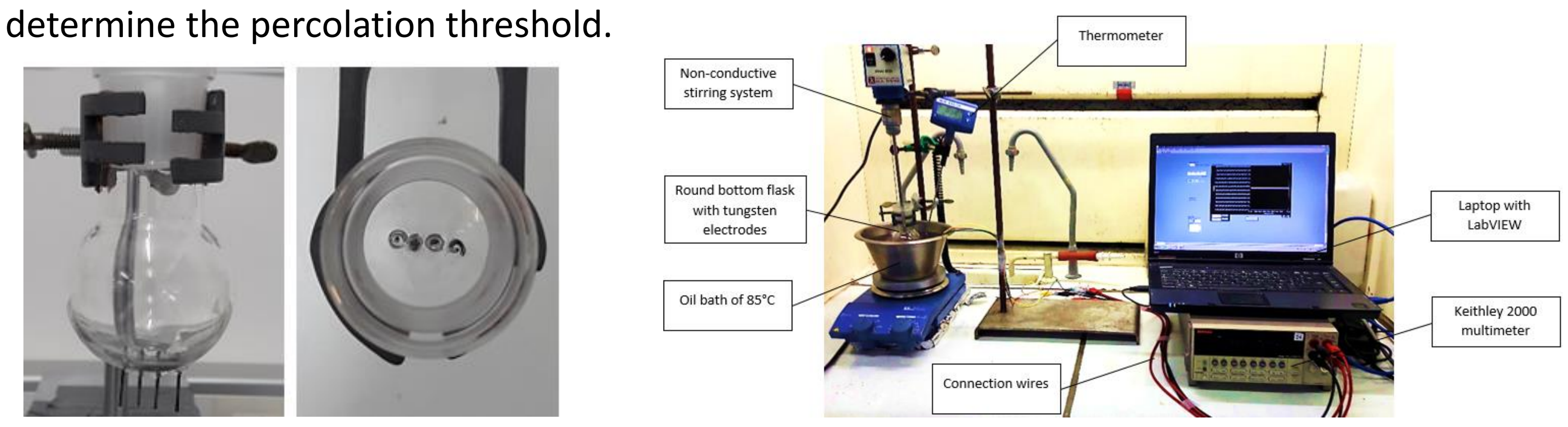

Figure 1: Design of round bottom flask with built-in tungsten electrodes (left) ; measurement set-up (right)

Table 1 shows the finally obtained volume percentage of each conductive filler in a PDMS-matrix and its corresponding blend resistance, which is also graphically represented in Figure 2.

Table 1: Filler load and corresponding resistance

CONDUCTIVE FILLER	FILLER LOAD (vol%)	RESISTANCE (Ω)
Carbon black	61.16	823097
Multi-walled carbon nanotubes	6.11	1763
Silver nanowires	2.07	No value
Silver microparticles	37.50	No value
Silver coated copper flakes	18.14	26.5
Carbon black & carbon nanotubes	35.91	73

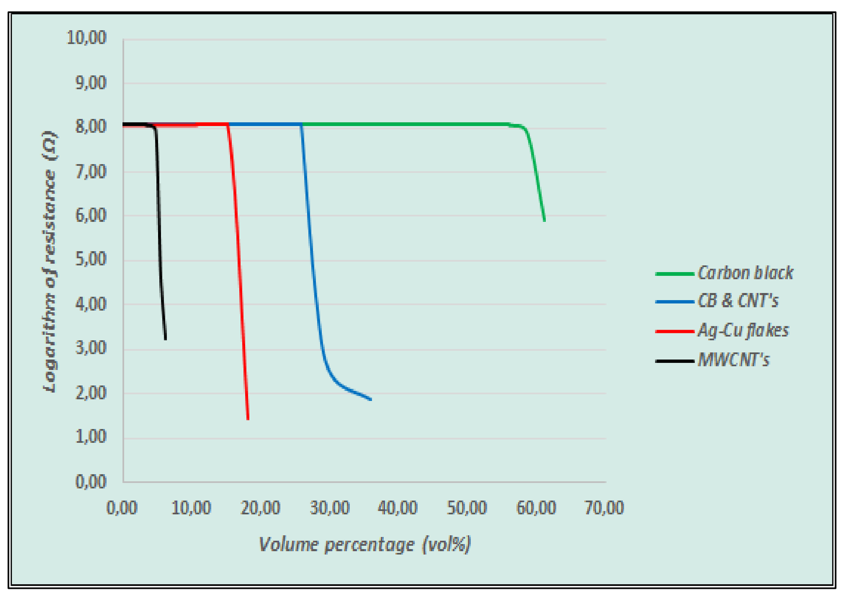

Figure 2: Comparison of percolation threshold

The percolation threshold of the silver nanowires and silver microparticles was not reached, while the other conductive fillers estimated or passed their percolation threshold.

CONDUCTIVE SELF-HEALING ELASTOMER

The self-healing elastomeric matrix was synthesised from fatty acid derivates (Pripol1017), urea and dipropylene triamine (DPTA). In the first step, Pripol1017 was condensed with DPTA and randomly branched oligomers were obtained. After a purification step, the oligomers reacted with urea to form a self-healing elastomer (Figure 3) [2].

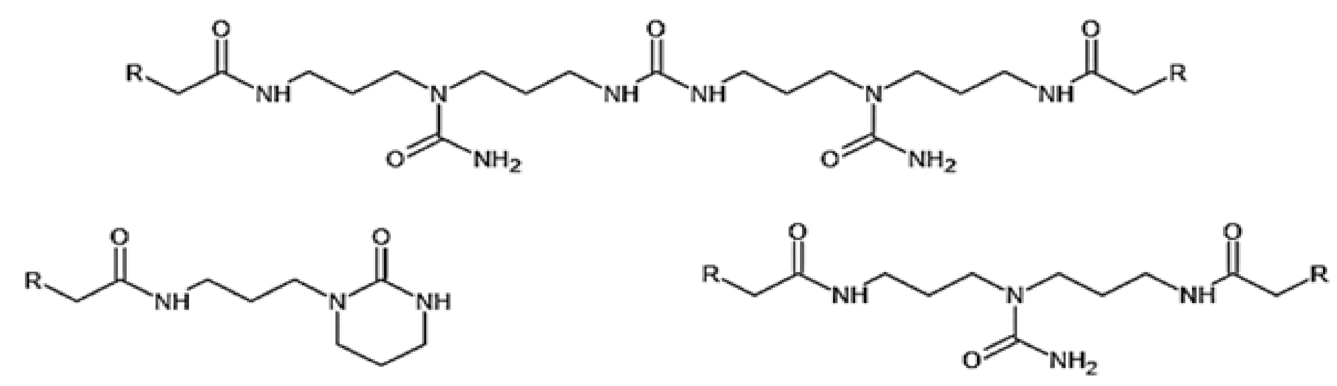

Figure 3: Structural formula of self-healing mixture

The addition of conductive fillers into the self-healing matrix reduced its electrical resistance, but increased the blend viscosity. Due to this, chloroform was used to dissolve the self-healing elastomer into a printable blend.

MECHANICAL & ELECTRICAL CHARACTERISTICS

The correlation between the electrical resistance and extensibility of the cured PDMS-inks with conductive fillers was investigated (Figure 4).

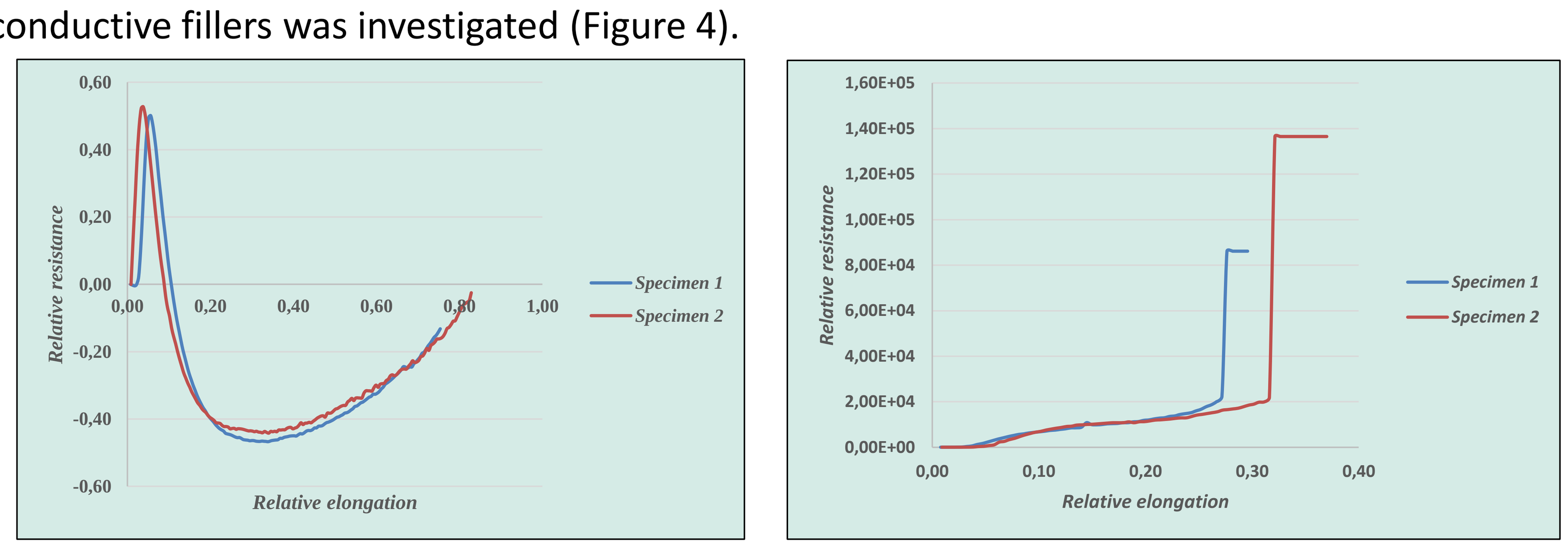

Figure 4: Relative resistance as a function of relative elongation: cylindrical particles (left) ; spherical particles (right)

CONCLUSION

The percolation experiments show that cylindrical particles (e.g. MWCNT's) have a lower percolation threshold than spherical particles (e.g. carbon black). Moreover, the blend resistance of metal fillers is smaller than carbon-based particles. Furthermore, cured PDMS-layers with cylindrical particles do not lose any conductivity while stretching (until fracture) in contrast to PDMS-layers with spherical particles, whose resistances increase as a function of the elongation. Finally, the integration of conductive fillers into the self-healing elastomeric matrix results in a printable ink by the addition of chloroform.

[1] M. Park, J. Park and U. Jeong, "Design of conductive composite elastomers for stretchable electronics," *Elsevier*, 2014.
[2] D. Montarnal, P. Cordier, C. Soulié-Ziakovic, F. Tournilhac and L. Leibler, "Synthesis of Self-Healing Supramolecular Rubbers from Fatty Acid Derivates, Diethylene Triamine, and Urea," *Journal of Polymer Science*, 2008.

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