



# The effect of bending loading conditions on the reliability of inkjet printed and evaporated silver metallization on polymer substrates

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## ABSTRACT

Systematic investigation of the effect of tensile and compressive cyclic bending strains on the mechanical reliability of inkjet printed and evaporated conductive silver lines on polyethylene naphthalate substrates is presented. With the help of a new bending test apparatus it is shown that cyclic tensile, compressive, and mixed tensile-compressive bending strains result in different amounts of induced mechanical damage in printed silver lines. In contrast, evaporated silver lines with the same geometry show no dependence on the type of strain. A detailed comparison of the fracture mechanisms in printed and evaporated silver is given using scanning electron microscopy and focused ion beam analysis.

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## 1. Introduction

The market for flexible, printed and large area electronics is expected to grow rapidly within the next years. Low-cost and high speed production, light weight and mechanical flexibility are the main factors which grant large market opportunities for flexible electronic technologies. The main challenges on the way to mass production are to find appropriate material combinations and fabrication methods which enable the realization of the above mentioned advantages. With the growing degree of maturity of the fabrication technologies the question of proper reliability testing becomes more and more important. The most demanding mechanical reliability test for flexible electronics is the bending test. For example, each of the layers of a multilayered flexible display structure must keep its functionality after repeated bending [1]. Comprehensive characterization of the bending stability and clear comparison of the results obtained by different groups are difficult due to two main reasons. First, there are many fabrication parameters, such as ink chemistry, printing methods, sintering parameters, substrate surface modifications, etc., which have a drastic influence on the mechanical stability [2–4]. Second, there is no standardized bending

test, which enables similar test conditions for different flexible electronics components. So far there are several conceptually different bending tests proposed by different groups [5–12]. The existing cyclic bending tests suffer from several disadvantages. Most of the referenced tests have a low throughput since only one sample can be tested at once. Sample clamping is often not trivial; in some test configurations a sample is already in a bent state when mounted or additional adhesive layers may be required to fix the sample. Application of both tensile and compressive bending strain is either not possible or requires complicated setups. The bending curvature is often not clearly defined and additional calculations are required to deduce the actual bending radius.

To foster the continuous development of fabrication methods and optimization of materials for flexible electronics a unified bending reliability test method and a systematic bending reliability characterization of different flexible thin film systems are required. In this paper a new tool for fast and reproducible characterization of the bending reliability of flexible electronic components is presented. With the help of the new bending apparatus the influence of tensile, compressive, and mixed tensile-compressive bending on the damage formation in inkjet printed and evaporated silver lines is investigated. Detailed analysis and comparison of the fracture mechanisms in conductive printed and evaporated silver lines using scanning electron microscopy (SEM) and focused ion beam (FIB) is also presented to further elucidate the fracture mechanisms.

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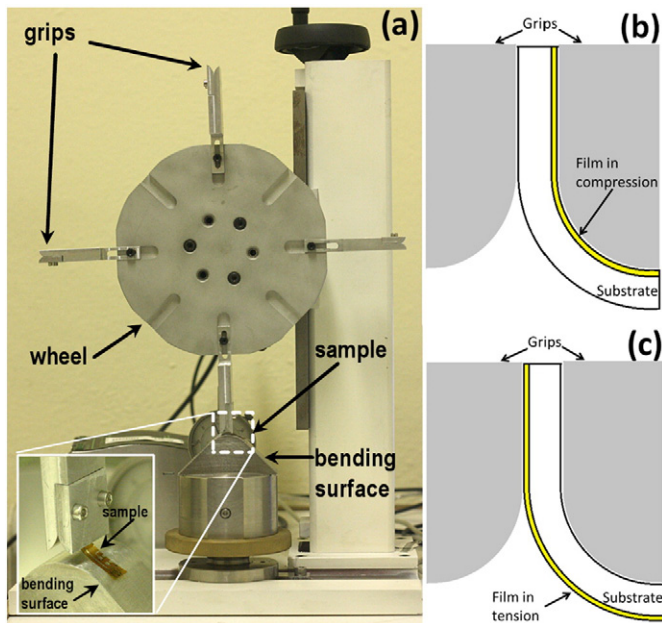


Fig. 1. Photograph of the FLEX-E-TEST bending demonstrator test setup (a). The inset at the bottom left shows the enlarged view of the sample in the bent state. Schematic presentation of application of compressive (b) and tensile (c) strain in the FLEX-E-TEST apparatus.

## 2. Experimental

The bending tests were performed using a newly developed technique called FLEX-E-TEST (Fig. 1). The samples are clamped at one end in grips which are fixed to a motor-driven wheel. Each grip has curved ends with the curvature radii corresponding to the required bending radius to be applied during the test. During rotation the samples sequentially come into contact with the bending surface and bend to the corresponding radius. The FLEX-E-TEST technique provides several important advantages in comparison to existing bending tests. The demonstrator equipment (Fig. 1a) allows one to test up to 8 samples simultaneously with the possibility to apply different bending radii as well as both tensile and compressive strains to different samples. Sample mounting is fast and straightforward, there is no need to use any adhesives or additional sample supports. The bending radius is strictly defined for each sample by the curvature of the grips, no recalculations or adjustments are required. Another important feature which is not provided by any of the existing bending methods is that between the bending–unbending cycles the sample stays in the flat state during the time needed for one full rotation until the next contact with bending surface. Such regime of the mechanical loading closely simulates potential “real-life” usage of a flexible electronic device. Finally, using the motor controller software it is possible to apply more complicated automated loading profiles by changing speed, rotation direction or by introducing additional waiting times at specific wheel positions during testing.

In this study three different bending loading conditions are considered. Within the first condition (Fig. 1b) repeated compressive strain is applied to the sample, which means that the line or film is on the inner surface when the substrate is bent around the grip. The second loading condition, tensile bending (Fig. 1c), corresponds to the line or film on the outer surface of the substrate during bending. The third condition, mixed bending, is a combination of tensile and compressive bending when the direction changes after every 10 cycles. The rotation speed was set to 1 Hz for all tests. All tests were performed using 5 mm bending radius which corresponds to 1.3% of cyclic strain.

All samples were prepared on 125  $\mu\text{m}$  thick polyethylene naphthalate PEN (Teonex® Q65HA) substrates purchased from Teijin DuPont Films.

Inkjet printed silver lines with a thickness of approximately 700 nm and a width of 500  $\mu\text{m}$  and the length of 40 mm were fabricated using a PIXDRO LP50 printer equipped with a FujiFilm Dimatix Spectra S-Class SE-128 piezoelectric print head. As silver ink the CCI-300 Ag-nanoparticle based ink formulation from Cabot Corporation with a nominal solid content of 19–21 wt.%, and a nominal viscosity of 11–15 mPa·s (at 22 °C) was applied. The size of the silver nanoparticles ranges between 30 nm and 100 nm. Substrate temperature was kept at 60 °C during printing at a resolution of 800 dpi. After printing the line pattern, the samples were cured at 150 °C for 2 h in a drying oven. The evaporated silver lines with the same dimensions were thermally evaporated in a vacuum coating unit at a base pressure lower than  $1 \cdot 10^{-6}$  mbar and a deposition rate of 10  $\text{\AA}/\text{s}$  using an appropriate shadow mask for structuring. The purity of the silver source was at least 99.95% and no substrate heating was performed during the deposition. The resistivity of inkjet printed silver lines was determined to be about  $2.1 \times 10^{-7} \Omega \cdot \text{m}$  which is approximately an order of magnitude higher than the resistivity of the evaporated lines. The average grain size of the evaporated silver was 620 nm. The residual stresses within the lines measured by X-ray diffraction and the  $\sin^2\psi$  method were about 100 MPa tensile for printed lines and slightly above 150 MPa tensile for the evaporated lines.

In order to analyze the evolution of the damage in the lines a series of tests were performed with different numbers of bending cycles between 500 and 50,000. For each maximum cycle number two to three different samples were tested in order to exclude the effects of possible fabrication defects and to provide enough statistical data. The amount of mechanical damage was characterized by means of the linear crack density which was calculated by taking a series of SEM micrographs in the middle of the line after a given number of bending cycles. For each micrograph the number of cracks per unit length was deduced and then averaged.

## 3. Results

The evolution of the crack density with the cycle number for three different bending loading conditions is shown in Fig. 2a for inkjet printed silver and in Fig. 2b for evaporated silver lines on PEN. It exhibits qualitatively similar behavior for both printed and evaporated lines: the crack density increases to approximately 10,000 cycles and saturates thereafter. However, the inkjet printed samples exhibit a strong dependence of saturation crack density on the loading type. The lowest crack density at a saturation level of 0.08 cracks/ $\mu\text{m}$  (corresponding to an average crack spacing of 12.5  $\mu\text{m}$ ) is observed for the compressive bending. Under tensile bending the crack density increases faster with the cycle number and the saturation value reaches 0.12 cracks/ $\mu\text{m}$  (corresponding to an average crack spacing of 8.3  $\mu\text{m}$ ) which is 50% more than in the compressive bending case. The mixed bending condition appears to be the most fatal for the printed silver lines. The corresponding saturation crack density is almost two times higher than after compressive bending but also the initial slope of the curve is significantly steeper.

The evaporated lines (Fig. 2b) show no statistically significant difference between the various loading types. In comparison to inkjet printed lines, the initial slope of the curves is steeper which indicates faster propagation of the cracks with the cycle number. The saturation crack density for all three curves lies around 0.13 cracks/ $\mu\text{m}$  which is very close to that of printed lines under cyclic tensile bending strain.

The SEM micrographs of the printed and evaporated lines are shown in Fig. 3. Inkjet printed silver lines exhibit long cracks, indicated by arrows, running between the nanoparticles and oriented perpendicular to the loading direction. Between the cracks the film remains undeformed, which is common for brittle fracture usually observed in nanocrystalline materials. In contrast, evaporated lines show typical ductile failure which includes propagation of

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