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SEM observations of a metal foil laminated with a polymer film

Nasir Mehmood[b], Eskil Andreasson[a, b, *], Sharon Kao-Walter[b,c]

[a]Tetra Pak® Packaging Solutions, SE 221 86, Lund, Sweden
[b]Dept. of Mech. Eng., Blekinge Institute of Technology, SE 371 79, Karlskrona, Sweden
[c]Fac. of Mech. & El. Eng., Shanghai Second Polytechnic Univ., 201209, Shanghai, China

Abstract: A thin metal foil laminated on a polymer film usually fracture at higher strains than its corresponding freestanding material layer. On the contrary the polymer film can be observed to fracture at smaller nominal strains when laminated. This is due to the strain localization induced by the created localised neck and plastic deformation in the metal foil. A significant reduction of the "gauge length" of the polymer film is observed locally. This scenario prevails if the adhesion is sufficiently high to prevent delamination to grow between the layers. The newly created gauge length is in the order of two times a metal foil thickness if the adhesion is very strong, leading to local high stress and low strains measured globally. However, this effect is not due to the brittleness of the material or shift of mechanical properties during lamination. During stretching, large deformations are observed in the moderately ductile and strain-hardening polymer film. Tensile failure (boundary conditions and geometrical effects) of polymer laminates has been observed to be governed by two mechanisms demonstrated in Fig. 1. below. In the first case, the polymer film forms a neck and is deformed locally where the metal foil has fractured and ruptures at a small strain (I). In the second case, the delamination is grown and the polymer deforms and delocalizes the strain to a substantial larger area (II). In some cases the laminated material creates multiple necks and the metal film ruptures at several positions and thus deforms at larger strains. All these observations have experimentally been demonstrated by using scanning electron microscopic (SEM) micrographs.

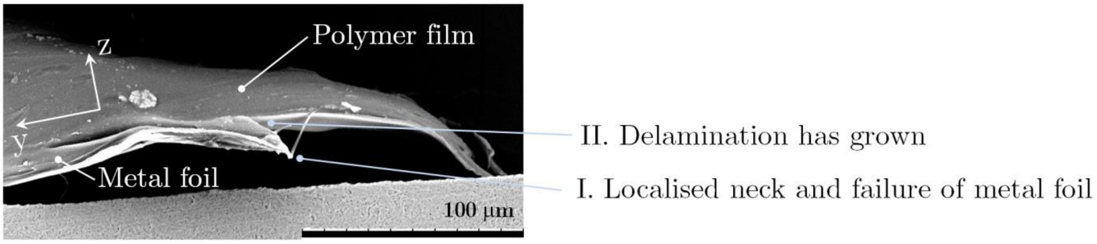

Fig. 1. Localisation, debonding and fracture of a one-sided laminated metal foil stretched in the y-direction. Strain-hardening and necking has prevailed in the polymer film and the metal foil has been exerted to localised plastic deformation. The micrograph is taken through the thickness direction.

Keywords: Adhesion; metal; micro-mechanisms; polymer; strain localization.

* Corresponding author. Tel.: +4-646-363-269;
E-mail address: eskil.andreasson@tetrapak.com

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1. Introduction

Demands for sustainable packages and increased shelf life in the packaging industry are pushing technologies to create a better barrier material that can extend the product life and reduce the waste. In packaging industries, fracture mechanical tensile testing has become very popular. This is used in the development of new material layers used as a member in the packaging material. These experimental testing are a pre requisite to determine the performance and mechanical material properties i.e. stiffness, ultimate strength, ductility, plasticity and Poison's ratio. This study covers the deformation mechanics involved in the non-linear elastic and elasto-plastic materials, describing the ductile behaviour of materials subjected to large mechanical loading and their eventual failures. Inelastic behaviours, large strains, geometric instability, strain localization and bifurcation from homogenous mode of deformation to severe deformation is investigated and analysed using various experimental techniques i.e. uniaxial tension testing, scanning electron microscope (SEM) analysis. During these experiments it has been observed that a reduction in the cross sectional area, caused by a defect or void can result in the localization of stresses. Many researchers has previously highlighted the importance of fatigue crack initiation due to repeated loading and micro cracks Koehl (1984), Koehl (1986), Hale (2001), Denny and Kitz(2005). Haibo and Spaepen (2000) have studied the relation between length scales of the microstructure and the physical properties. A freestanding metal film (Aluminium) accommodates local elongation as the ruptured halves moves apart. Suo et al. (2005) has showed in several articles that the low ductility of a metal film is a result of local thinning. A bonded metal film (2000) on a polymer substrate can't accommodate local elongation so a delocalization in the strain field is observed due to the substrate. This delocalization leads to larger strains before rupture.

2. Materials

The investigated materials in this study are: a 6.3µm thick moderately ductile metal foil produced by aluminium and a strain hardening highly ductile polymer substrate produced by a 20µm low density polyethylene (LDPE) film. The studied LDPE material, showed an increase in the slope of stress-strain curve at large strains indicating the strain hardening phenomenon, recently studied by Jönsson et al. (2013).

3. Experimental procedures and observations

3.1 Scanning Electron Microscope (SEM) procedure

SEM is a very powerful equipment for making very high quality pictures with a lot of detailed material information. It is very important to understand the cutting and preparation of the specimens before studying them in SEM. The point where the sample is taken is also very important to know for highly ductile material that can have variation, in the ductility of stretched samples at different strains and geometric instabilities due to deformation. The Fig. 2 is showing the complete procedure to prepare the samples for SEM.

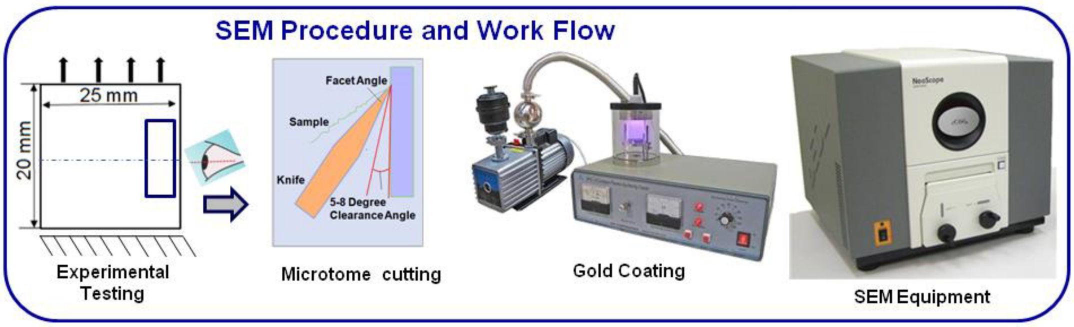

Fig. 2. SEM sampling procedure at Tetra Pak, Lund

Samples have been taken from one of the fracture pieces to look at the cross-section. Observations have been made in the thickness direction of the material. Using a microtome cutter LEICA RM2255, 70µm thin samples are prepared at a cutting angle of 5-8 degree for microscopic examination to get 3D micrographs, shown in Fig. 2. The sharp blades have been used to cut the thin samples. In order to prevent the charging effects, an additional conductive gold layer was sputtered onto the samples prior to the observations in the SEM equipment. The gold layer is used to achieve the optimal resolution by eliminating the charging effects. G. Rochat et al (2004) has studied these charging effects of gold coating on polymers. Fracture surface and material morphology is observed in a NEO scope JCM 5000 Scanning Electron Microscope after the specimens are completely unloaded. It has been possible to observe the details in the size of 2µm with this procedure.

3.2 Experimental observations from tensile tests

For accurate and realistic modelling of the Aluminium-foil and its laminate structure, the individual material properties are studied. Strain hardening phenomenon has been observed during uniaxial tension testing on the LDPE film. Large strain deformation behaviour is observed for steeply hardening polymer substrate. The thin polymer film, after initial yielding (between 3-4 %), strains are plastic and localization accompanied with necking has occured. The film stretches and a local thinning is observed. Experimental results for metal to polymer laminate have been focused in this study. Experimental results are presented in Fig. 3, showing the results for a weakly hardening material (Aluminium metal) laminated to a steeply hardening polymer substrate. At small strains weakly hardening material carries high stress than steeply hardening material. Point 'a' indicates the complete breakage of weakly hardening material and points 'b'-'f' showing the constant force necking and strain localization at larger strains. A strain hardening phenomenon and an increase in the strain hardening modulus is observed at point 'g'. A full stretching is observed at point 'h' and after this point, material breaks in a brittle fashion. The delamination front and step by step delamination process is described by black dotted line in Fig. 3. At high strains, the strain hardening polymer showed steeply increasing stress behaviour.

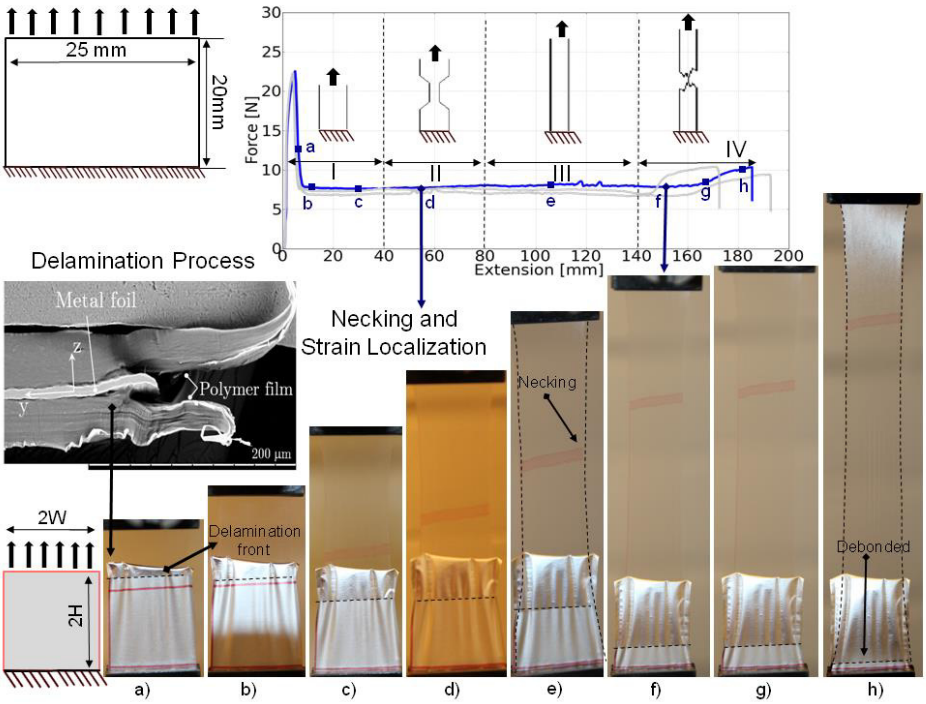

Fig. 3. Continuum material results for Al-Laminate, Specimen dimensions 25*20. Strain hardening in a steeply hardening polymer film (I) Initial plastic strain (II) Neck stabilizes and local thinning (III) Strain hardening (IV) Eventual rupture.

The fracture strain for moderately or low extensible materials is very small as shown in the Fig. 3. Aluminium carries higher stresses at small strains while LDPE has comparatively lower stresses at the same strain levels. Aluminium fractures at low strains, rendering the LDPE to constantly deform at lower stresses and start debonding from aluminium. A large irreversible deformation is observed for ductile polymers. The LDPE material exhibit stable neck and the molecular orientation provide a mechanism for hardening that dominates at large strains as shown in graph of Fig. 3. from I-IV. Molecules are aligned parallel to the stretching direction. At this moment, the film starts getting strain hardening and an increase in the strain hardening modulus. Asaro and Rice (1977) have modelled the plastic flow as rate-insensitive and localization is found to be possible only if the plastic hardening modulus for the slip system has fallen to a certain critical value. Experimental evidence has shown substrate bonded metal film is hardening at large strains. During the tensile extension, appearance of wrinkles has been observed. These wrinkles are appearing due to the compressive stresses present in the lateral direction perpendicular to the direction of applied load triggering out of plane bonding in the thin structure. Due to volume conservation law, the visual necking or lateral compression has motivated the creation of wrinkles. The stresses are assumed to be concentrated in the centre crack edges. The crack propagation rate is also quite slow due to the presence of ductile and steeply hardening polymer substrate.

3.3 Strain Localizations and Delocalization

SEM observations and microfilming has shown strain localization in ductile metal films by a single cross-slip as represented in the micrograph a) in Fig. 4. The plastic flow in the metal film showed a bifurcation from a more or less homogenous mode of deformation to a concentrated one in a narrow shear band. J.P. Bardet (1990) has reviewed that strain localization theory specifies the conditions for which shear band emerges within the uniformly stressed and strained materials. After detection of existence of strain localization, shear band orientation and velocity field inside the shear band at onset of localization can be determined from strain localization theory. The limitations in the theory of strain localization have been solved using the SEM experiments, where, emergence of shear band, development of shear band and thickness of shear band is calculated. Huang (2009) described the limitations of length scale in the continuum mechanics to calculate the thickness of shear band using theory. Debonding of the Aluminium in the laminate caused the strain localization at an early stage during the deformation process. Debonding and strain localization will progress simultaneously as described by Dietmar and Thomson (2011).

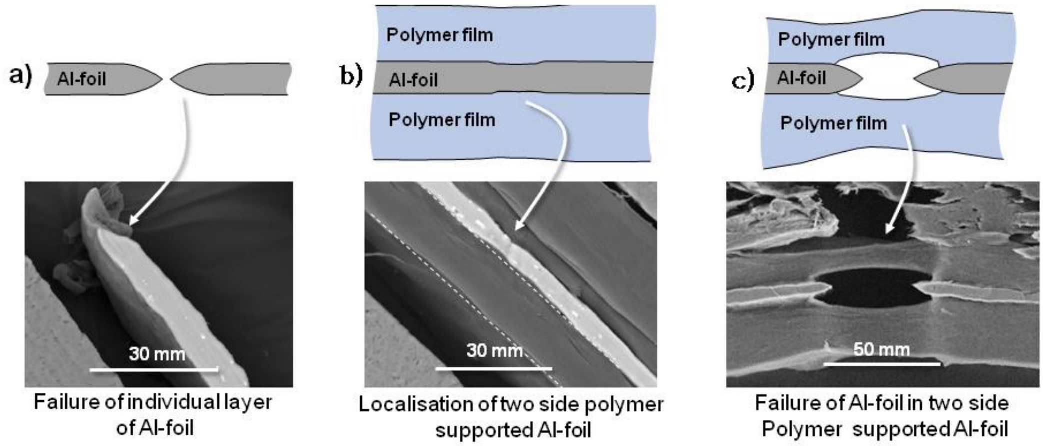

Figure 4.SEM Illustration and micrographs of the deformation mechanism of a freestanding and laminated metal foil a) Freestanding metal foil rupture b) necking and localization at small strains in laminate c) Strain localization and chisel edge formation in metal-polymer laminate.

For aluminium laminate material with good adhesion the surface sustained large strains without rupture. Aluminium laminate has been observed to propagate the shear band at small load breaking the material into pieces at different strains as shown in Fig. 4c). A material in the necking region is capable of supporting much larger stresses than outside the neck. The fracture mechanical relevant microstructure for ductile fracture is normally characterized by the specific heterogeneous composition. The rupture strain of the metal film is sensitive to adhesion to the substrate as presented by Yong et al. (2005). The functional performance of composite material depends on the cohesive strength of the metal film and its adhesion to the polymer substrate.

When the applied strains are small laminate deforms uniformly but as the stress increases the non-uniform deformation develops. Hutchinson and Tvergaard (1980) has analysed the sensitivity of the shear band growth to material imperfections for ductile materials. However the conditions for bifurcation into a shear band will be discussed in the next article in more detail.

4. Results & Discussion

The laminate has shown different level of adhesion and debonding. For a good adhesion the aluminium laminate acted as a stiffer material and for bad adhesion the laminate has shown smaller stiffness and deformation was also low due to early debonding. It has also observed that variation in the level of adhesion forces the material to show a multiple necking phenomenon. Carpinteri & Paggi (2007) found that a competition between crack trajectories can take place due to bi-material interface and loading direction. Fig 5b) below is showing the specimen with bad adhesion of aluminium-metal i.e. release of aluminium layer from LDPE, the films started to get thinner and cracks are initiated at higher strains. Once a crack is formed by deformation or by necking, it can locally amplify the stress, thereby decreasing the ultimate strength and rendering the aluminium-foil, more susceptible to breakage by imposition of single large stress. For moderately flexible materials it is observed that adhesion level is also moderate. Bulacu (2008) showed that on account of local entanglement and molecular chains the polymer molecules form a complex three-dimensional network. Under tensile loading, microscopic deformation initially comprise of rotations and stretching of single chains segments. Subsequently, debonding of intermolecular crosslink and straining as well as rupture of chains may occur. In the studied laminate fracture process, a similar situation is accompanied by the formation of micro voids. With increasing deformation, voids localizes in thin zones perpendicular to the microscopic loading directions.

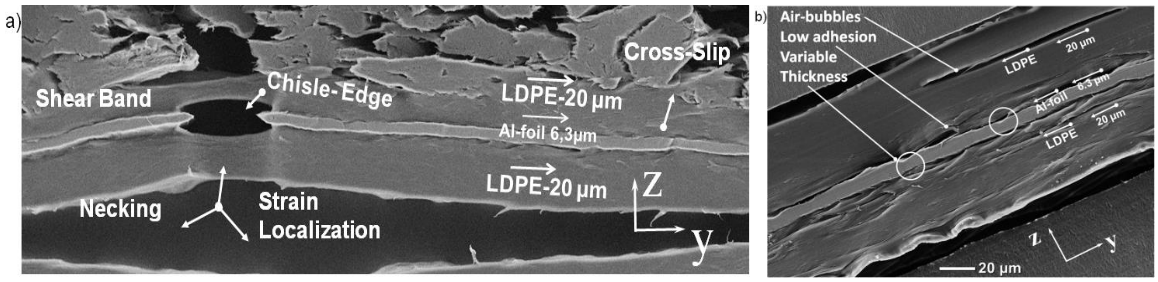

Fig. 5. Localization of plastic flow into one or multiple shear bands is observed for studied aluminium laminate. (a) Shear band or Strain localization and debonding in aluminium laminate; (b) Multiple necking leading to multiple shear bands in aluminium laminate.

An x-shaped shear band formation is also observed during the severe deformation of the laminate as shown in Fig. 5a). This subsequent deformation led to strong localization as explained by Suo et al (2005). The relevance for shear banding in the laminate is that they precede failures as the extreme deformations within the shear band lead to intense damage or failure. During the process of deformation, the metal film forms a chisel edge leading to a shear band with continuous deformation as indicated in Fig. 5a). On the other hand, this continuous deformation also forms multiple necking leading to multiple shear bands as shown in Fig. 5b). Necking and thinning is also observed for polymer substrate on both sides. This concept of shear band formation is used as a key to understand the failure in the metal film laminated with ductile polymer substrates. During uniaxial tensile testing, a complete rupture of material structure is performed to observe the behaviour of involved material layers of different thicknesses. Boundary conditions and geometrical effects of the polymer laminate has shown that the failure occurs when the polymer forms a neck and is deformed locally after aluminium foil has ruptured at small strain. It is also observed that he grown delamination has caused the polymer material to deform and delocalize to large strains.

5. Conclusion

A modification and improvement has been adapted to the traditional SEM sample preparation and micrographs. With the new technique a 3D effect is retrieved in the visualization of the SEM sample. This enhanced the possibility to better evaluate the fracture surfaces in SEM. Micromechanical fracture behaviour of moderately ductile metal film on a highly ductile polymer substrate has been shown using SEM. Necking and strain localization or shear band formation behaviour is simulated following the observation from SEM micrographs. The findings from the virtual model and the technique to successfully implement the real behaviour from SEM micrographs have not been the focus in this article. Micrographs for the shear band formation have been shown using the developed SEM technique. The developed experimental technique has helped to capture the involved deformation and fracture mechanisms during the complete loading of the specimen. The video and SEM micrographs of the deformation behaviour after yielding has enabled to understand the necking at an almost a constant force and delamination of the Aluminium foil from the polymer laminate. A virtual model describing the complete deformation process i.e. yielding, necking and delamination has started working. SEM observation has shown that it is hard to get a uniform thickness for Aluminium foil and the thickness variation is present through the length. SEM results of Aluminium-laminate have helped to observe the multiple necking and several shear bands. Fracture mechanical uniaxial tensile test results have shown that,

- Aluminium-laminate doesn't have a catastrophic failure as compared to stiffer polymer laminate. Crack propagation rate is significantly low for Aluminium-laminate.
- Strain localization is observed as Aluminium gets necking. Adherent polymers have shown thinning.
- Effects of debonding due to strain localization have been observed. Metal film and polymer are debonding at high strains.
- Delocalization is observed for materials that exhibit good adhesion.
- The studied technique has been a valuable input for the development of a virtual model for strain localization and shear band.

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