Analysis of AE Signals during Scratch Test on the Coated Paperboard

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Abstract

The deformation and fracture behavior of ink-printed layers on clay-coated paperboards were estimated using the scratch tester with the acoustic emission (AE) method. The designed scratch tester was custom-designed made for the paper. To investigate the boundary effects of coated and printed layers on the fiber matrix, two kinds of paperboards were prepared. Scratch tests were carried out using three types of machined spherical indenters with an AE transducer attached on the surface of the paperboard. The shape of compressed marks was observed by the laser microscopy. The measured AE signal was analyzed by amplitude and peak frequency and related to the deformation and fracture behaviors. The detected AE generation was divided into two zones: one corresponded to the micro-crack generation and the other was the generation of delamination. This AE generation behaviors varied with the indenter tip radius.

Keywords: Scratch test, paperboard, coated layer, micro-crack, delamination

Introduction

Paperboard has received much attention since it is easily and cheaply recycled. Further, in order to meet modern packaging needs, the coloring and texture have both been enhanced using different base papers and coating substances. Printed paperboard surfaces are damaged by scuffing and abrasions during careering operation in the shape forming and carton forming processes. The fracture behavior of painting surfaces needs to be easily quantified in order to evaluate their mechanical properties. Scratching tests are available to evaluate the mechanical properties of coating substances. These tests have been used to evaluate the strength of adhesion between a hard membrane and a matrix such as a deposited film or plating layer on a metallic surface [1]. It has also been reported that the scratching test is useful for evaluating the mechanical properties of paperboards [2, 3]. However limited results were obtained regarding the strength of a coated layer or paper substrate, based on the compressive load or width of scratching indent. These results could not be used to completely evaluate the adhesion strength of the printing layer. Furthermore, the relationship between the adhesion strength and the fracture behavior is not clear, and is insufficiently understood to confirm the effect of indenter shape on the fracture behavior during the scratching process.

Acoustic emission (AE) is useful in detecting fracture behavior in dynamically deformed materials. It reveals AE signals, which correspond to the deformation and fracture behaviors during the paperboard cutting process [4, 5]. AE signals during the cutting, ruling and embossing machining have been collected and analyzed in the same manner [6-9]. However, AE signals from surface fractures such as scuffs and abrasions have not been widely studied.

In this study, scratching tests were examined on two types of paperboards, a black-ink layer printed surface and a white-clay layer coated surface, with three scratching indenter diameters under constant scratching rates to investigate the abrasion phenomenon and the fracture behaviors within the very thin depth region of 50 µm. AE signals were detected in the respective
scratching tests, and the compressed traces were observed using microscopy. These signals and microscopic images were synchronized and verified to recognize the fracture behaviors of coated layers.

**Experimental**

*Materials:* The fundamental structure of the paperboard is displayed in Fig. 1. Paperboard is composed of a pulp fiber structure matrix and clay structured coated layer having a color printed layer and varnish. To investigate the boundary strength of the printed paperboard, two pattern paperboards were prepared for the scratch test. One was conventional coated paperboard of 230 g/m² nominal basis weight and 0.3 mm thickness. Specimens were cut to 100 mm in length and 15 mm in width. The second was printed paperboard of carbon black pigment with a varnish layer. It was the same size and weight of unprinted paperboard.

![Fig. 1 Fundamental structure of the printed paperboard.](image)

*Estimation of mechanical properties:* The machining strength of the base paperboard was examined using the tensile test along the machining direction (MD) and cross direction (CD) following the JIS P8113 specification. To estimate the friction coefficient between the surface of the paperboard and the indenter material, the dynamic friction test (horizontal method) was carried out with a specially designed machine. Figure 2 shows a schematic illustration of the frictional system.

*Scratching test and AE detection system:* Figure 3 shows a schematic illustration of scratching test configuration and AE system using the universal testing machine. The automatic X-stage was attached to the lower crosshead, and the specimen was placed upon the SUS630 steel face-plate embedded on the X-stage. Indenters were made of SKD tool steel and were shaped with...
tip radii of 0.05, 0.1 and 0.5 mm. The spherical shaped indenters were attached below the upper crosshead with a load cell. The universal testing machine was operated by lowering the upper crosshead at a constant speed of 0.001 mm/s in the vertical direction and the X-stage was synchronized at a constant speed of 0.1 mm/s in the horizontal direction. The applied load was measured over the duration of indenter contacts with the surface for a penetration depth of 50 µm.

The AE waves were detected using AE sensors attached to the sides of the indenter. The AE detection and analysis system is shown in Fig. 4. The AE sensor has a resonance frequency of 220 kHz, and signal processing was operated with a 10 kHz high-pass filter and a 400 kHz low-pass filter. These examinations were performed at 296 K, 50% RH in the thermo-hygrostat room, and were repeated ten times. The scratching traces of the width and depth were observed using laser microscopy. The images of the scratching were synchronized to the time scale of the scratching and AE signals, and the elastic waves were related to the fracture behaviors. The scratching mark profiles were also traced from the surface texture measurements.

Fig. 3 Schematic illustration of the scratching test with the AE analysis system.

Fig. 4 Schematic illustration of the AE analysis system.

Results and Discussion

Characteristics of the paperboard: To evaluate the structure of the printed paperboard, the cross sectional view was observed using a laser microscope. The observed result is shown in Fig. 5. A paperboard is composed with a pulp fiber matrix, clay layer, printed ink layer and varnish. The clay is adhered on the pulp matrix with 10 to 20 µm thickness, and ink layer has a few µm thick-
Figure 6 shows the surface conditions on each paperboard surface for (a) non-coated surface, (b) surface of the coated layer with clay and (c) printed surface with black ink (Carbon black) and adhered to the varnish on the ink layer. In this observation, the surface roughness could also be evaluated. The results are shown in Fig. 7. On the non-coated surface, many fibers are adhered and arrayed in one direction. The surface roughness became large. In the clay layer coated surface, as the space of the jointed fiber structure is filled with clay, the surface roughness become less than the non-coated surface. On the other hand, when the printed surfaces are covered with black carbon ink and varnish, the surface roughness depended on the printed ink elements, thickness and flatness. The surface roughness value of the printed paperboard improved much more than other surfaces. This indicates that, if the adhesion strength between the coated and printed ink layer could be estimated with some mechanical method, the printing condition could be evaluated on every printed paperboard. The scratch test and AE method was applied so this evaluation could be realized.

Fig. 5 Sectional view of the paperboard using a laser microscope.

Fig. 6 Observed top surface using a laser microscope.

Strength of paperboard: To evaluate the strength of the paperboard, the machine direction (MD) and cross-machine direction (CD) tensile tests were carried out on the coated and printed paperboard, using the tensile machine following the JIS-P8113 specification. The results are shown in Table 1. As the difference in tensile strength values between coated and printed paperboard for each tensile direction was small, the strength of the printed layer on the coating layer could not be evaluated using the tensile test.
Table 1. Tensile strength of coating and printed paperboard on MD and CD.

<table>
<thead>
<tr>
<th>Material</th>
<th>Coated</th>
<th>Printed (black ink)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MD</td>
<td>CD</td>
</tr>
<tr>
<td>Tensile Strength (MPa)</td>
<td>36±3</td>
<td>19±1</td>
</tr>
<tr>
<td></td>
<td>MD</td>
<td>CD</td>
</tr>
<tr>
<td>Tensile Strength (MPa)</td>
<td>36±2</td>
<td>19±1</td>
</tr>
</tbody>
</table>

Table 2 Results of friction test on coated and printed paperboard.

<table>
<thead>
<tr>
<th>Material</th>
<th>Coating</th>
<th>Printed (black ink)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dynamic friction coefficient</td>
<td>0.24~0.25</td>
<td>0.23~0.24</td>
</tr>
</tbody>
</table>

The results of the friction coefficient tests are shown in Table 2. The friction coefficient of printed paperboard is a little less than that of the coated paperboard. It was concluded that the surface roughness slightly affected the friction coefficient. As the scratch test could be carried out under small friction force, the deformed depth and width value seem to be almost independent to the surface conditions. Hence, when the indentation force is sufficiently small, the AE signal would be detected under a low noise level without any effects of compressed inside layer of paperboard.

**Scratch test:** Figure 8 shows a typical AE signal with the vertical forces on the same time scale. The corresponding scratching trace image on the clay layer is shown in Fig. 9 with an indenter radius of 0.1 mm.
**Coated paperboard:** On the clay layer, AE signals were generated from the moment of indenter contact and continued during the scratching process. In the observed compressed surface, many cracks could be detected inside the scratched surface. This indicates that the AE signal resulted from the friction behavior of the clay layer surface and the flaking behavior of the boundary region of the fiber layer. To confirm the separation behavior of the coating layer from fiber matrix, a one-dimensional compression test was carried out with the same tensile machine on the printed layer. Figure 10 shows the penetrated hole shape after the compression test. Figure 10(a) and (b) respectively show the results when using a 0.5 and 0.1 mm radius tip. In Fig. 10(a), it is assumed that the hole surface was evenly deformed, but in case of the smaller tip radius, 10(b), some micro cracks could be detected on the hole wall. This shows that the printed and clay layer did not break during the compression process in the large tip. An AE signal could not be observed during these tests, but was detected during the scratch tests. Figure 11 shows the typical AE signal and vertical forces on the same time scale, and the corresponding scratching trace images on the printed layer are shown in Fig. 12 with an indenter radius of 0.1 mm.

**Fig. 10 Compressed hole with a tip radius of (a) 0.5 and (b) 0.1 mm.**

**Fig. 11 Typical relation between AE generation and vertical forces during the scratch test on the printed paperboard. (tip radius of 0.1 mm)**
Fig. 12 Observed compressed trace image during scratch test on printed paperboard.

For indentation depth below 40 µm, no AE signal was observed (region S1). In the larger scratch depth region of 40 to 100 µm, AE was continuously generated (region S2). In this region many micro-cracks could be observed on the surface edge of the scratch mark, as shown in Fig. 12. When the scratched depth became larger than 100 µm (region S3), AE signal generation increased abruptly. It is assumed that the AE behavior corresponded to the separation of the printed layer from the coating layer and the fracture of fibers.

Effects of indenter tip radius: The compressed trace shape of the scratched surface changed with the indenter tip radius. To investigate the effects of tip radius, three indenters were machined for this experiment, and they were compressed to a depth of 50 µm (50 sec). The scratching tests were carried out on a printed surface with carbon black pigment ‘black’, using the indenters with a tip radius of (a) 0.5 mm, (b) 0.1 mm, and (c) 0.05 mm, respectively. Relationship between the scratch depth, AE amplitude, and vertical force, is shown in Fig. 13 for each indenter tip radius. Every vertical force increased proportionally with the penetrating depth, and values in the clay layer were always less than the printed layer. This indicates that the clay layer was harder and stronger than the printed layer. Figure 13(a) shows the result of the largest indenter radius (0.5 mm). The AE signal could not be observed under this penetration depth, and only the compressed shape was impressed on the surface. On the compressed trace, no micro-cracks were observed by the estimation system. This indicated that the indenter penetrated into the printed layer smoothly in this large depth region.

In Fig. 13(b), when the indenter tip radius was 0.1 mm, the force curve increased with scratching depth more smoothly than the 0.5 mm radius case. Further, the first AE signal detected, corresponding to micro-crack generation, could be observed around 40 µm, as shown in the SEM observation result. The observed AE amplitude existed between the region of 30 to 40 dB. In the smallest radius case (0.05 mm), the force curve trended upwards, and AE signal was detected around a depth of 20 µm. The amplitude of the AE signals was observed in the wider region to be from 30 to 60 dB (compared to the 0.1 mm radius). Micro-cracks and delamination were detected in the same compressed region. Comparing the results of Fig. 13(b) and (c), the cracks are generated by a vertical force of over 0.3 N and the corresponding AE signal could be observed in the amplitude range of 30 to 40 dB. On the other hand, delamination was also detected on the compressed trace surface. It appears that micro-cracks occurred during
delamination under this tip radius condition. Comparing Figs. 13(c) with (b), AE signals near 40 dB were not detected, and delamination did not occur in Fig. 13(b). It is obvious that the greater AE signals can be attributed to the occurrence of delamination. It was also confirmed that deformation without fracture could not be detected in the AE signals in Fig. 13(a). As a result, the AE signal amplitude on the coated paperboard can be classified into three ranges from the scratching test: elastic compressive deformation at less than 28 dB (A1), crack initiation in the range of 28 to 40 dB (A2) and the occurrence of delamination above 40 dB (A3).

It is assumed that the three deformation behaviors would be generated by:

1. Elastic deformation of the entire structure.
2. Plastic deformation of the printed surface layer after a yield point.
3. Break-down of the surface and clay layer.

The generation point of these deformation behaviors varied with indentation tip radius. The deformation behavior of advanced indentation surface area changed with the applied load surface area, which depended on the stress concentration. On the other hand, a notable number of AE signals are observed in Fig. 13(a). These AE signals can be classified by their amplitude, greater than 40 dB and less than 30 dB. In Fig. 13(b), the AE signals decreased drastically, and the upper level signals did not appear on this time scale. Despite the high vertical force, AE signals could not be detected, as shown in Fig. 13(c). The detected AE signal corresponded to accelerated motion phenomena such as brittle crack generation and delamination. As the results of AE generation behavior on Fig. 13(a), the AE signal generation level were divided into three region in comparison with the crack and delamination. The schematic AE generation behavior on small tip radius of 0.05 mm, is shown in Fig. 14. In the early stage (A1), there were no AE signals but only deformation was observed. Cracks generation occurred in the lower AE amplitude region (A2), and the delamination occurred higher amplitude region (A3), almost same indenter compressed depth area.

![Fig. 14 The classification of the AE signals corresponding to fracture behaviors.](image)

**Summary**

To evaluate the deformation and fracture behaviors of printed paperboard surfaces, the scratch test was carried out and the AE method was used simultaneously. The main results are summarized below.

1. The strength of the surface layer on the printed paperboard could not be evaluated using the normal mechanical method.
2. On the clay coating layer, many cracks occurred after a very low applied load, and many AE signal were detected at the same time.
3. During the compression test, micro-cracks were detected in the penetrated wall when a small indentation radius was used, but no AE signal was observed.
4. When an indentation radius of 0.1 mm was used AE signal generation began at 40 µm, which corresponded to the occurrence area of micro-cracks. Abrupt AE generation occurred over 100 µm, which was attributed to delamination.
5. The deformation and fracture behavior of surface printed layers changed with the indentation tip radius, and the generation of AE signals also varied with changes in fracture behavior. It was considered that stress concentration effects in the advanced deformation surface area, depended on the micro-crack occurrence phenomena.
6. The AE signals could be detected and categorized into three regions in order of amplitude, and on the same time scale corresponded to compressive deformation, crack initiation, and delamination. It was revealed that the AE method provides an effective evaluation of the printed layers on paperboards.

References