

Depth analysis for obtaining information about chemical composition and chemical bonding at different depths in a solid sample is a useful technique for characterizing and evaluating materials. For the preparation, an oblique cutting technique with a microtome¹⁾ is usually used to improve the resolution in the depth analysis of materials, especially polymers and polymer composites. This technique allows us to increase the sample depth on an oblique section that is obtained by cutting the sample surface at a very small angle. Thus, analyzing the oblique section produces a higher depth resolution. However, this high-precision small-angle cutting has demanded careful manipulation and many hours of processing.

We have developed an oblique cutting machine that makes it possible to produce, both precisely and rapidly, an oblique section with a small angle on a sample surface.²⁾ This machine consists of a sample stage with a goniometer, a cutting tool, and a cutting tool driver, as shown in **Fig. 1**. The most notable feature of the cutting method is the deep cutting into the sample surface using the cutting tool (**Fig. 2a**), and subsequently making an oblique section at an angle α along the direction that perpendicularly intersects the cutting direction (**Fig. 2b and 2c**). The processing time, including sample setting and cutting, is only five minutes and the reproducibility of the oblique cutting angle is less than 0.02 degrees at a set-up angle of 1.0 degree.

The developed machine was applied to the depth analysis of organic pigments in multi-layered coating films. An oblique section of the coating film in both an initial sample and an outdoor-exposed sample,

were measured in the depth direction at 100- μm steps by means of attenuated total reflection infrared micro-spectroscopy. The oblique cutting angle was 1.0 degree and the expansion ratio of the sample depth was 57 times. Under these conditions, the depth resolution was 1.7 μm in the longitudinal direction. The depth profiles of the amide group characteristic of the organic pigment are shown in **Fig. 3**. It was found that the amount of organic pigment in the outdoor-exposed sample decreased near the surface layer of the base coat. This result confirms that this machine is a useful and powerful tool for the high-resolution depth analysis of polymers and polymer composites.

References

- 1) Esaki, Y., et al. : 34nenkai Kouen Youshishuu (in Japanese), (1984), 362, Jpn. Soc. Anal.
- 2) Masao, T., et al. : 53nenkai Kouen Youshishuu (in Japanese), (2004), 362, Jpn. Soc. Anal.

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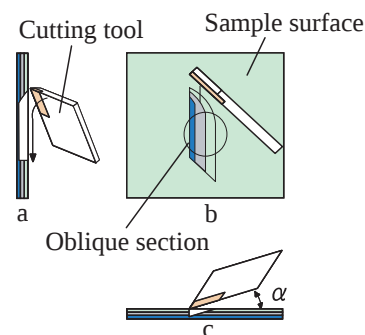


Fig. 2 Schematic diagram of the cutting method.

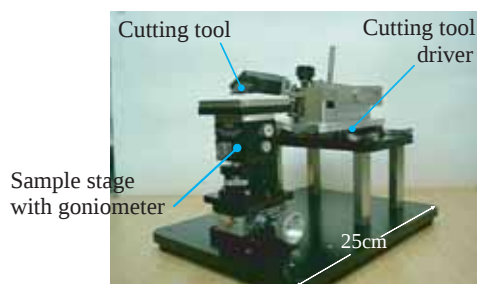


Fig. 1 Appearance of the oblique cutting machine developed.

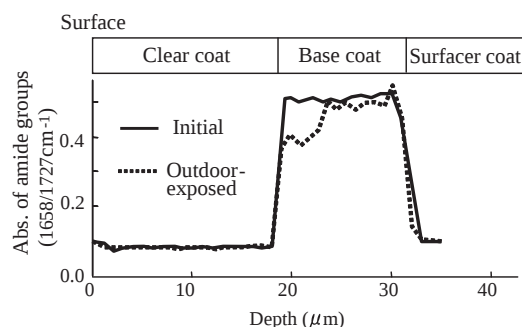


Fig. 3 Depth profiles of an organic pigment in the multi-layered coating films.