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DINAMIC MECHANICAL ANALYSIS OF PAPER SAMPLES

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# ABSTRACT

A clamping technique was worked out for dynamic mechanical analysis of thin cellulose and paper sheets. The technique has been applied in the study of original and aged papers.

#### INTRODUCTION

Among the methods suitable for the investigation of paper aging, there are several ones reflecting molecular physicochemical changes. On the other hand, mechanical properties related to the structure of the material - are also very important from the aspect of practical applications. A number of methods - usually standardized - are on hand to test mechanical characteristics of paper. Yet, none of these methods is free of shortcomings concerning sensitivity or reproducibility, etc. Censequently, the introduction of new test methods is still needed.

One of the possibilities is dynamic mechanical analysis /DMA/, describing sample stiffness and dissipation as functions of temperature. Changes of these properties are related to the internal motions of polymer molecules or their segments.

The present paper is an account on DMA investigation of papers. Some other results of our research program on cellulose and paper aging have been published elsewhere [1].

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# MEASURING TECHNIQUE

The investigations were carried out on a DuPont 981 Dynamic Mechanical Analyser measuring the resonant frequency and damping of the system consisting of the sample and the instrument arms [2,3]. Most solids are rigid enough to be tested as a sheet or rod. Soft materials /e.g. thermosets / may be measured with the aid of support techniques [4-6]. Buckling deformation of thin films can often be avoided with horizontal mounting [4]. However, thin cellulose and paper sheets / of 0.1 mm thickness or below / could not be tested in the usual way as both vertically and horizontally mounted samples showed the buckling mode of deformation. The support techniques were not expected to be suitable either, because of rather small changes to be detected. Therefore, a sample geometry different from a flat sheet was sought to prevent buckling deformations.

In the measurements reported here, a special sample clamp assembly was applied, providing an arched sample cross section / for details see ref. [7] /.  $5^{\circ}$ C/min geating rate was chosen, and a nitrogen flow purged the sample space.

# RESULTS AND DISCUSSION

Papers of different quality were treated at 100°C in air of 50% relative humidity; the DMA curves of original and aged samples were recorded. Figures 1 and 2 show the behaviour of two sorts: a newsprint and a writing-printing paper free from wood-pulp.

The curves of resonant frequency and damping resemble the curves of synthetic polymers to some extent, as they contain regions of frequency and damping. The resonant frequency of paper samples showed a gradual decrease up to  $230-250^{\circ}C$  and a fast, change above this range. Each damping curve had a dereasing section to  $80^{\circ}C$ , and a shoulder between 240 and  $300^{\circ}C$  followed by a fast decrease. The changes near  $300^{\circ}C$  can be related to the glass transition and the onset of thermal

decomposition. Beyond the changes listed above, the aged samples showed a broad damping maximum or shoulder in the  $100-200^{\circ}C$  range, where original papers had a constant damping level.



Fig.1. Frequency and damping curves of a newsprint./----/ Original, /---/ after lo days of accelerated aging, /..../ after 30 days of accelerated aging. Fig.2. Frequency and damping curves of a writing-printing paper free from wood-pulp. /---/ Original, /---/ after 6 hours of accelerated aging.

This effect may probably be used in the characterization of aged papers, howewer, its relation to the differences of structure has not been elucidated so far.

#### CONCLUSIONS

The results of investigations on aged papers prove that the

sample mounting technique worked out by the authors [7] can be applied in dynamic mechanical analysis of paper sheets or other samples of similar characteristics.

The DMA curves may be sensitive to the changes of paper structure caused by aging. The authors are studying the possibility of quantitative evaluation of the frequency and damping curves obtained by means of the clamping technique applied here.

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