Institute of Paper Science and Technology
Atlanta, Georgia

IPST Technical Paper Series Number 792

The Relationship Between ZD Ultrasonic Stiffness and Beta Formation

J.P. Gerhardstein, P.H. Brodeur, L.D. Walsh, and J.F. Waterhouse

May 1999

Submitted to
Journal of Pulp and Paper Science

INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY
PURPOSE AND MISSIONS

The Institute of Paper Science and Technology is an independent graduate school, research organization, and information center for science and technology mainly concerned with manufacture and uses of pulp, paper, paperboard, and other forest products and byproducts. Established in 1929, the Institute provides research and information services to the wood, fiber, and allied industries in a unique partnership between education and business. The Institute is supported by 52 North American companies. The purpose of the Institute is fulfilled through four missions, which are:

- to provide a multidisciplinary education to students who advance the science and technology of the industry and who rise into leadership positions within the industry;
- to conduct and foster research that creates knowledge to satisfy the technological needs of the industry;
- to serve as a key global resource for the acquisition, assessment, and dissemination of industry information, providing critically important information to decision-makers at all levels of the industry; and
- to aggressively seek out technological opportunities and facilitate the transfer and implementation of those technologies in collaboration with industry partners.

ACCREDITATION

The Institute of Paper Science and Technology is accredited by the Commission on Colleges of the Southern Association of Colleges and Schools to award the Master of Science and Doctor of Philosophy degrees.

NOTICE AND DISCLAIMER

The Institute of Paper Science and Technology (IPST) has provided a high standard of professional service and has put forth its best efforts within the time and funds available for this project. The information and conclusions are advisory and are intended only for internal use by any company who may receive this report. Each company must decide for itself the best approach to solving any problems it may have and how, or whether, this reported information should be considered in its approach.

IPST does not recommend particular products, procedures, materials, or service. These are included only in the interest of completeness within a laboratory context and budgetary constraint. Actual products, procedures, materials, and services used may differ and are peculiar to the operations of each company.

In no event shall IPST or its employees and agents have any obligation or liability for damages including, but not limited to, consequential damages arising out of or in connection with any company's use of or inability to use the reported information. IPST provides no warranty or guaranty of results.

The Relationship Between ZD Ultrasonic Stiffness and Beta Formation

J.P. GERHARDSTEIN, P.H. BRODEUR, L.D. WALSH, AND J.F. WATERHOUSE

Institute of Paper Science and Technology, 500 10th St. N.W. Atlanta, GA 30318

A comparison study between ZD longitudinal ultrasonic specific stiffness and beta formation has been made to investigate possible relationships between ZD specific stiffness and grammage variations. Stiffness measurements were obtained using a newly developed ultrasonic instrument capable of generating two-dimensional maps of out-of-plane paper specific stiffness properties, including soft-platen thickness and traveling time. A beta formation instrument was used to gather two-dimensional maps of grammage. Measurements were performed on 10 x10 cm laboratory and pilot linerboard samples with different formation indices. Grammage, soft-platen thickness, traveling time, and specific stiffness data were compared point by point at the 10-mm spatial resolution level. Also, coefficients of variations for the various parameters were compared. Results indicate that localized measurements do not correlate. However, relationships exist among coefficients of variations when samples are globally considered.

INTRODUCTION

Determination of paper stiffness properties using nondestructive ultrasonic techniques has been an on-going research area for several years. The main goal is to develop real-time, on-machine techniques to monitor the mechanical behavior of paper during the papermaking process and, hence, provide the necessary information for process control and end-use performance optimization [see for example refs. 1-3]. On the basis that paper is considered an orthotropic material, nine elastic stiffness constants are needed to fully describe its elastic behavior [1]. All nine constants can be measured [4, 5]. Of particular interest here is the thickness-direction (ZD) longitudinal constant C_{33}. This constant is known to correlate to ZD tensile strength and provide a nondestructive means of predicting the internal bond strength [1]. In a recent study involving C_{33} and Z-toughness to evaluate bond strength, both test methods were successful in their ability to measure improvements in bonding created by refining, wet pressing, and polymeric strength aid [6]. In the context of on-machine monitoring of the mechanical behavior of paper during papermaking, the most important attribute of C_{33} is its insensitivity to in-plane fiber orientation. This makes it an essential component of a strategy to decouple in-plane stiffness dependency upon fiber orientation and processes such as wet pressing and wet straining [1].

The determination of C_{33} is obtained through the measurement of the ZD longitudinal specific stiffness, i.e.,

$$C_{33}/\rho = (h/t)^2 \tag{1}$$

where ρ is the soft-platen apparent density of paper, equal to the grammage (G) divided by the soft-platen thickness (h), and t is the traveling time of the bulk ultrasonic wave propagating in the

thickness direction of paper. In effect, $C_{33} = (G\,h)/t^2$, and it is assumed that G, h, and t are independent parameters. It is of particular interest to investigate how h, t, and C_{33}/ρ vary locally and how they relate to small-scale grammage variations. Also, it is of interest to examine global relationships between these parameters. This is the essence of the present work, which is centered on the use of a beta formation instrument and a newly developed out-of-plane ultrasonic instrument capable of recording two-dimensional maps of soft-platen thickness and traveling time and, hence, maps of C_{33}/ρ.

There has been some related work exploring the relationship between density and specific stiffness. On a global basis, good correlation was observed between these parameters [7, 8]. However, since they are both sensitive to thickness (density $\propto 1/h$ and specific stiffness $\propto h^2$), one may expect some degree of correlation. In any case, the relationship appears to be independent of how the sheets are densified, i.e., by refining or wet pressing, for sheets which have been dried under full restraint. Therefore, on a local basis, we might expect to see some degree of correlation depending on local density variability, but it is not expected to be as strong as on a global basis.

The impact of formation on C_{33} was studied as well [9]. It was found that as formation deteriorated, there was little impact on the average global C_{33}. However, there was a very significant increase in C_{33}/ρ variability with increased deterioration in formation. Furthermore, the level and variability of in-plane specific stiffness appeared to be independent of formation as expected, because of the large difference in the scales of measurement. Therefore, in the present study, as formation deteriorates an increase in ZD specific stiffness variability is expected to be seen.

A description of the ultrasonic mapping instrument is first introduced, followed by a brief description of the beta formation instrument. Then, the details of the experimental work are presented. Finally, results obtained for different linerboard samples are reported and discussed.

EXPERIMENTAL

Out-of-plane Ultrasonic Stiffness Instrument

A new instrument was developed at IPST for measuring out-of-plane stiffness properties using ultrasonic waves. This instrument substantially differs from an earlier prototype instrument [10] in its ability to provide fully automated 2-dimensional maps of out-of-plane stiffness using different transducer configurations. A schematic of this instrument is shown in Figure 1.

At the heart of this instrument is a pair of ultrasonic transducers. These are wide bandwidth, commercially available PZT (Lead Zirconate Titanate) transducers with 20-mm diameter active surfaces. The lower transducer is rigidly attached to the base of the instrument, and the upper transducer is mounted to a motorized translation stage via a steel shaft in a low-friction linear bearing. Longitudinal transducers at 1, 5, and 10 MHz, as well as shear transducers at 1 MHz, are available and are easily interchanged. Removable cylindrical- and conical-shaped polystyrene delay lines are attached to the active face of each transducer. A 0.76-mm (0.030-inch) thick, soft (30 Durometer Shore A) neoprene rubber tip is epoxied to the face of each delay line used with the longitudinal transducers to improve coupling with the paper sample. Delay lines with 3-, 10- and 20-mm diameter tips are available, allowing sample testing with different spatial resolution.

The upper transducer assembly is counterweighted such that it has a neutral balance in the linear bearing. A loading mass is placed on top of the transducer assembly to provide a 50-kPa loading

pressure between the transducers. A different mass is available for each diameter delay-line tip to produce the correct loading pressure.

An LVDT (linear variable displacement transducer) thickness gauge is used to measure the thickness of the sample during testing. Because the neoprene tips and loading pressure used in this instrument are the same as specified in TAPPI test method T-551 [11], the thickness measured is similar to soft platen. However, the delay-line tip area and some other details of the mechanism are different from T-551; hence, the thickness measurements are not rigorously soft platen.

The transmitting transducer is driven by a 400 V_{p-p} single cycle sine wave with a duty cycle of 0.2%. The receiving transducer signal is routed through a preamplifier and then to a 100-MHz, 8-bit A/D board to digitize the received signal. Both the preamplifier and the A/D board have variable level gains to control the amplitude of the received signal. The LVDT gauge is read using a 16-bit A/D board and has a theoretical thickness resolution of 0.065 μm, but the LVDT driving electronics are noise-limited to around 0.3 μm. The signal excitation electronics, as well as the high- and low-speed A/D boards are completely contained inside a PC computer. The motorized translation stage and X-Y table are also computer controlled and have a positioning resolution of 0.1 μm.

Before testing, an ultrasonic reference wave is recorded on an 8-μm-thick aluminum foil shim. This reference wave is used to eliminate the delay time of the electronics during testing. The 8-μm shim is also used as a calibration point for the LVDT thickness gauge. The slope for the LVDT calibration is determined by stepping the motorized translation stage up in ten 120-μm

steps and reading the LVDT voltage after each step. These 10 points are then fit to a straight line, and the slope is extracted for the LVDT calibration. The slope is combined with the 8-μm shim reading to compute a linear calibration for the LVDT.

To test a sample, the instrument may be configured to allow either the operator to position a sample by hand between the transducers or the computer to position a sample using the X-Y table and perform a mapping of the sample properties. Maximum sample size for the X-Y table is 24 cm square, and step size is operator selectable down to 1 mm. During testing, the motorized translation stage lowers the upper transducer until the sample is pressed between the two transducers. The linear bearing prevents the translation stage from applying any force directly to the transducer-sample contact area; hence, the contact force is produced entirely by the loading mass. While in contact, ultrasonic waves are passed through the sample by the transducers and the sample thickness is measured using the LVDT. Typically, several ultrasonic waves are averaged together to remove electronic noise from the measurements. A cross correlation technique described previously [12] is used to determine the time difference between the reference and test wave caused by the sample interposed between the transducers, i.e., the travel time through the sample. The thickness of the sample is then divided by the travel time to calculate the average velocity through the sample, and the average velocity is squared to compute the specific stiffness. The test wave then may be run through an FFT to analyze frequency information. The operator may enter an average grammage for the sample and a quasi-apparent local density (average grammage / local thickness) can be calculated. A more rigorous treatment considers the use of the local grammage $G(x,y)$ as obtained using a beta-ray gauge (described below), in which case the apparent local density $\rho(x,y)$ can be obtained. This allows the local stiffness constant

$C_{33}(x,y)$ to be determined on a point-by-point basis by multiplying the specific local stiffness by the apparent local density. Results are displayed in a graphical format, and statistics (average, maximum, minimum, standard deviation) are also calculated. Data can be saved to a file for further analysis.

Prior to beginning this study, the instrument went through a battery of tests to insure that it produced accurate and repeatable measurements. Metal shims and paper samples with known thicknesses between 55 and 1800 microns were used to verify the LVDT calibration. Excellent correlation ($R^2 > 0.99$) was found. Travel times of various samples measured using IPST's earlier prototype instrument [10] were compared with the new instrument. Good correlation was again found ($R^2>0.99$), but the best fit line had a slope of 0.90. The deviation from a slope of 1.0 was determined to be due to the older instrument working at a lower frequency (around 0.5 MHz), which, because of the dependence of frequency on wave velocity, corresponded to longer travel times. This was verified by driving the new instrument's 1-MHz transducers at 0.5-MHz, which then produced a travel time correlation plot with a slope of 1.0.

Repeatability tests were performed on several samples to look at the effect of recalibrating the instrument, removing the sample, and rotating the sample. All tests yielded high correlation coefficients ($R^2 > 0.98$) with the exception of rotation tests, which were lower ($R^2 \sim 0.90$-0.75) due to imprecise repositioning of the sample.

Beta Formation Instrument

Grammage variations were obtained using IPST's beta transmission/light transmission formation instrument. A schematic diagram of the instrument is shown in Figure 2. Samples of various sizes can be used and are held in place on the X-Y table using magnetic clamps. The X-Y table is programmed to transport the sample so that formation measurements can be made over a selected area. The instrument has the capability of making light transmission measurements in the wavelength range of 400-700 nm, in 20-nm increments. Mass density measurements are made using a 50-mCi Pm 147 source as a beta particle (fast electron) emitter. A beta particle detector is used to count the number of beta particles passing through the sample in a given time interval. In the present study, only beta formation measurements were performed. The following calibration equation was used to convert the particle counts to mass density (local grammage):

$$G_{(x,y)} = k\left[\ln(COUNTS_{air}/sec) - \ln(COUNTS_{(x,y)}/sec)\right] \qquad (2)$$

where $G_{(x,y)}$ is the grammage at location (x,y) (local grammage), $COUNTS_{(x,y)}/sec$ is the number of sample counts per second at location (x,y), $COUNTS_{air}/sec$ is the number of background counts per sec in the absence of sample, and k is the calibration factor obtained by considering the average grammage and the average number of sample counts per second.

TEST SAMPLES AND CONDITIONS

Six noncommercial linerboard samples were tested. They were chosen to represent different formations. Selected properties of interest are tabulated in Table I. All samples were uncoated boards made of virgin, unbleached, softwood fiber with no additives or fillers. Sample A was a single-ply liner made on IPST's Formette Dynamique. Sample B was made on a Web Former.

The Fourdrinier and C-Former samples were made on a pilot paper machine. The single-ply samples and the base sheet for the two-ply samples were made of pulp beaten to 600 ml CSF, and the top layer of the two-ply samples was made of pulp beaten to 400 ml CSF.

All samples were tested using both the beta formation tester and the 2-D mapping ultrasonic instrument. The samples were evaluated on the beta formation tester using a 1-mm square aperture and 5-second integration time. One measurement was made each millimeter in both the MD and CD directions over a 10 x 10 cm area to produce a grammage map. Ultrasonic measurements were obtained using the 1-MHz longitudinal transducers and 10-cm diameter neoprene-tipped delay lines. 2-D maps of soft-platen thickness and traveling time were made by taking measurements every centimeter in the MD and CD directions over an area of 10 x 10 cm. Every attempt was made to assure that both instruments were testing the same area on each sample. Because the grammage and thickness/traveling time measurements were performed using different spatial resolution levels (1 and 10 mm, respectively), the grammage measurements were resampled by computing the average over each 10 mm x 10 mm area to yield a 10 x 10 cm map with readings every centimeter in both directions.

RESULTS AND DISCUSSION

For the purpose of illustrating typical results, grammage maps are reported for two extreme cases: Sample A - good formation index and Sample F - poor formation index (see Table II). Figures 3 and 4 and Figures 5 and 6 summarize grammage (G) results for these samples at the 1- and 10-mm spatial resolution levels. Even though resolution is substantially lower at 10 mm, one can still observe formation features. Figures 7 to 10 report 2-D maps of soft-platen thickness (h), traveling time (t), specific stiffness (C_{33}/ρ), and elastic stiffness (C_{33}) for Sample A only.

The coefficient of variation was measured for all considered properties using a dimensionless formula put forward by Norman [15]:

$$\%CV(X) = \frac{\sigma(X)}{\overline{X}} \times 100\% \tag{3}$$

where $\overline{X}$ is the average property value and $\sigma(X)$ is the standard deviation of the local property measurements. This equation is considered to be the ideal equation for the modeling of formation [9] even though it does not provide any information on floc size distribution. The coefficient of variation for grammage and other measured properties is presented in Table II for all the samples. The coefficient of variation for grammage (formation index) was computed based upon resampled measurements (10-mm resolution). It is interesting to note that %CV for G, h, and t follow similar trends. Also, it is interesting to observe that %CV for traveling time is more constant across samples than %CV for grammage and thickness. As one might expect, %CV for specific stiffness and elastic stiffness are relatively larger because these properties are composite variables.

Figure 11 shows the relationship between the coefficient of variation for specific stiffness and. grammage (formation index) for all samples. The slope of the best fit line is nearly 1, indicating a possible relationship. However, the scatter of the data provides an R^2 of only 0.68, indicating that there is likely some other unmeasured variable in the relationship between specific stiffness and grammage.

Correlation calculations between the different properties on a local scale were performed as well and are shown in Table III. Apart from some evidence of weak correlation between thickness and traveling time ($R^2 = 0.51$), grammage does not correlate to thickness, traveling time, or specific

stiffness. This suggests, in the context of the present study, that the above properties are independent, which means that good formation does not necessarily imply good specific stiffness and vice-versa. In fact, if optimization of stiffness properties is the driving end-use performance factor for a given grade, a good formation index does not necessarily imply that stiffness properties will be optimized. More work is required with a large sampling of different commercial samples to verify this observation.

CONCLUSIONS

A new ultrasonic instrument capable of performing two-dimensional maps of out-of-plane paper stiffness properties was presented. Measurements were performed on different laboratory linerboard samples using 1-MHz longitudinal transducers at 10-mm spacing. Results were compared to mass density measurements obtained using a beta formation instrument. They indicate that correlation between local grammage and specific stiffness, thickness, and traveling time do not exist. However, a moderate correlation ($R^2 = 0.68$) was found between the global level of variability in grammage and specific stiffness. Hence, producing a sheet with good formation tends to reduce variability in stiffness but does not guarantee that the optimum stiffness has been obtained.

Future work will include reducing the test area to look at smaller-scale variations and expanding the study to include a wider range of samples.

ACKNOWLEDGMENTS

The authors would like to acknowledge the help of Ms. Floria Cheng from the University of North Carolina. Portions of this work were used by L.D. Walsh as partial fulfillment of the requirements for the M.S. degree at the Institute of Paper Science and Technology.

REFERENCES

1. BAUM, G.A., "Elastic Properties, Paper Quality, and Process Control," *Appita* 40(4):288-293 (1987).
2. HABEGER, C.C. and BAUM, G.A., "On-line Measurements of Paper Mechanical Properties," *Tappi J.* 69(6):106-111 (1986).
3. BRODEUR, P.H., JOHNSON, M.A., BERTHELOT, Y.H., and GERHARDSTEIN, J.P., "Noncontact Laser Generation and Detection of Lamb Waves in Paper," *J. Pulp Paper Sci.* 23(5):J238-J243 (1997).
4. MANN, R.W., BAUM, G.A., and HABEGER, C.C., "Elastic Wave Propagation in Paper," *Tappi J.* 62(8):115-119 (1979).
5. MANN, R.W., BAUM, G.A., and HABEGER, C.C., "Determination of All Nine Orthotropic Elastic Constants for Machine-made Paper," *Tappi J.* 63(2):163-166 (1980).
6. STRATTON, R.A., "Characterization of Fibre-Fibre Bond Strength from Out-of-plane Paper Mechanical Properties," *J. Pulp Paper Sci.* 19(1):J6-J12 (1993).
7. WATERHOUSE, J.F., "The Failure Envelope of Paper When Subjected to Combined Out-of-plane Stresses," *Proc. TAPPI Int. Paper Physics Conf.*, Kona, Hawaii, September (1991).
8. BITHER, T.W. and WATERHOUSE, J.F., "Strength Development Through Refining and Wet Pressing," *TAPPI J.* 75(11):201-208 (1992).
9. WATERHOUSE, J.F., "The Effect of Some Papermaking Variables on Formation," *TAPPI J.* 76(9): (1993).
10. HABEGER, C.C. and WINK, W.A., "Ultrasonic Velocity Measurements in the Thickness Direction of Paper," *J. Appl. Pol. Sci.* 32:4503-4540 (1986).
11. TAPPI Test Method T-551 "Thickness of Paper and Paperboard (soft platen method)," *TAPPI Test Methods 1996*, TAPPI Press, Atlanta GA (1996).
12. BRODEUR, P.H., HALL, M.S., and ESWORTHY, C., "Sound Dispersion and Attenuation in the Thickness Direction of Paper Materials," *J. Acoust. Soc. Am.* 94(4):2215-2225 (1993).
13. WALSH, L.D., "A Comparison Study Between High Resolution Out-of-plane Ultrasonics and Beta Formation," M.S. Report, Inst. of Paper Sci. and Tech., Atlanta (1997).
14. Baum, G.A. and Wink, W.A. "A Rubber Platen Caliper Gauge - A New Concept in Measuring Paper Thickness," *TAPPI J.* 66(9):131-133 (1983).
15. NORMAN, B. "The Formation of Paper Sheets," edited by J.A. Bristow and P. Kolseth, New York: Marcel Dekker, Inc. (1986).

TABLE I
SELECT LINERBOARD SAMPLE PROPERTIES

Sample Identification	**Forming Process & Sample Description**	**Average Grammage (g/m^2)**	**Wet Pressing Level**
A	Formette Dynamique (single ply)	230	Not Available
B	Web Former	222	Not Available
C	C-Former (single ply)	130	light
D	Fourdrinier (two ply)	191	heavy
E	Fourdrinier (two ply)	191	light
F	Fourdrinier (single ply)	134	light

TABLE II
COEFFICIENT OF VARIATIONS FOR DIFFERENT PROPERTIES
(10-MM RESOLUTION LEVEL)

Sample Identification	Grammage	Soft-Platen Thickness	Traveling Time	Specific Stiffness	Elastic Stiffness
A	1.54	2.45	4.63	5.59	7.64
B	4.40	4.36	5.28	11.39	11.27
C	4.77	5.33	5.88	7.61	10.06
D	6.98	8.14	6.60	11.85	13.07
E	7.23	7.86	6.80	10.37	13.35
F	7.24	7.84	6.82	12.33	13.19

TABLE III
DEGREE OF CORRELATION BETWEEN PROPERTIES ON LOCAL SCALE

Sample Identification	Thickness vs. Traveling Time	Grammage vs. Thickness	Grammage vs. Traveling Time	Grammage vs. Specific Stiffness
A	0.7477	0.0762	0.0503	0.0098
B	0.1169	0.0009	0.0074	0.0027
C	0.6060	0.0024	0.0001	0.0114
D	0.4936	0.0291	0.0560	0.0009
E	0.6163	0.1231	0.1646	0.0003
F	0.4660	0.2166	0.1508	0.0323
Average of All Samples	0.5078	0.0747	0.0715	0.0096

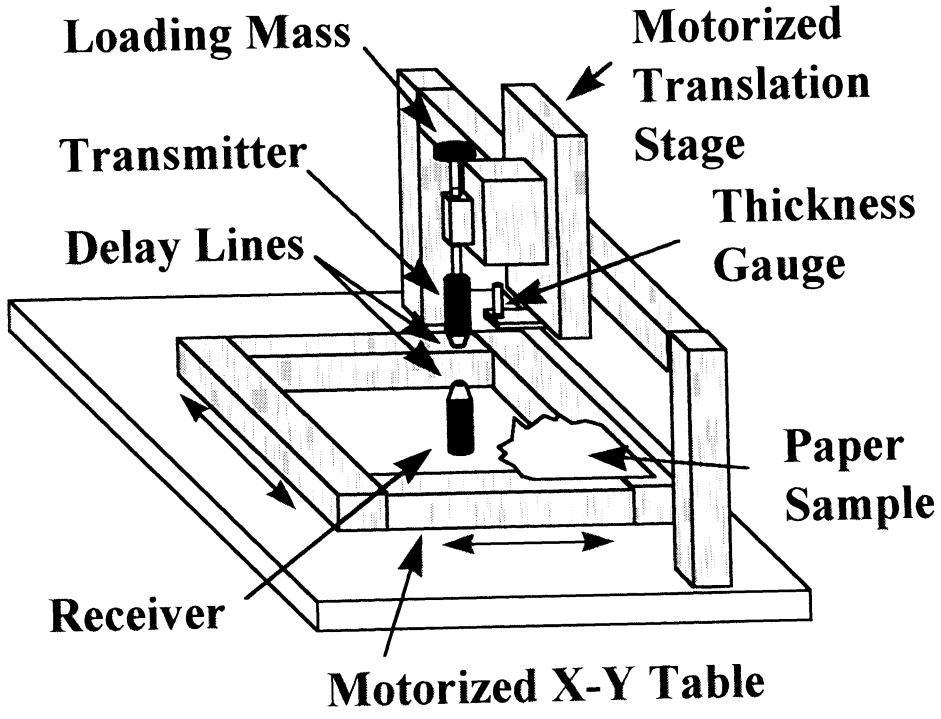

Fig. 1 Schematic of the out-of-plane ultrasonic stiffness instrument.

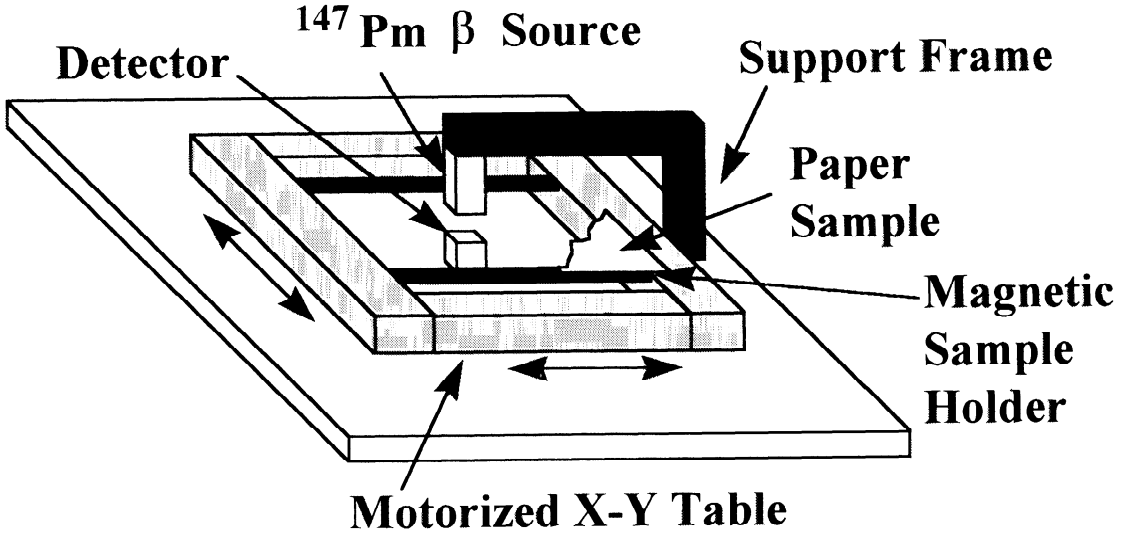

Fig. 2 Schematic of 2-D beta formation instrument used to record grammage variations.

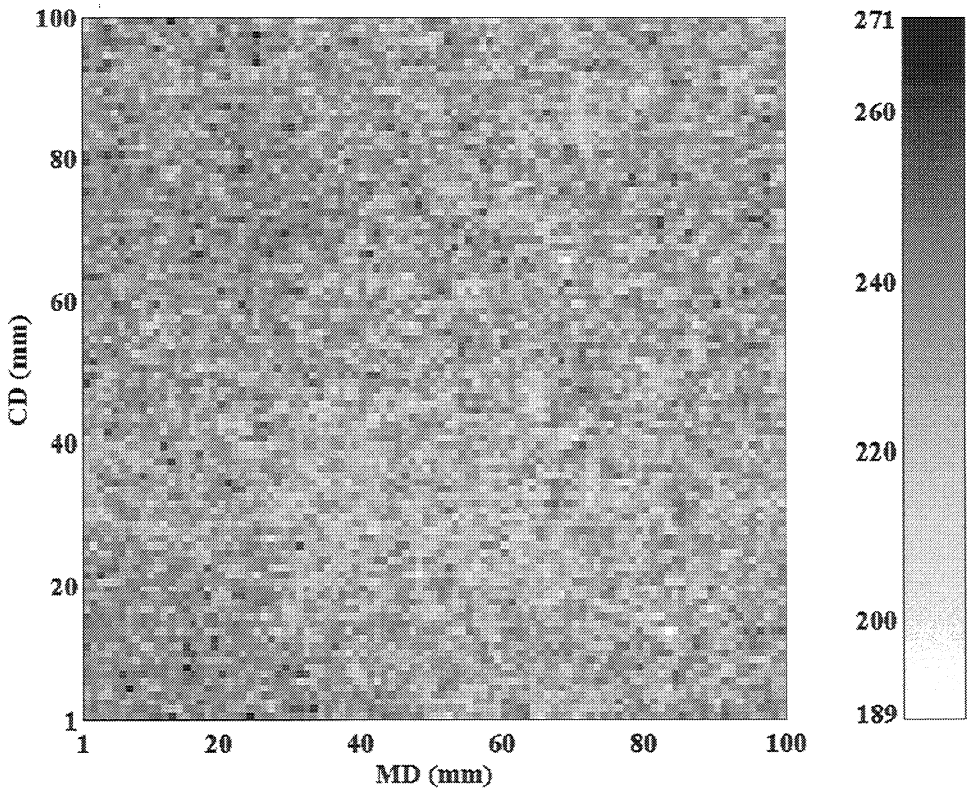

Fig. 3 Sample A grammage [G] map at 1-mm resolution (Z-axis in g/m^2).

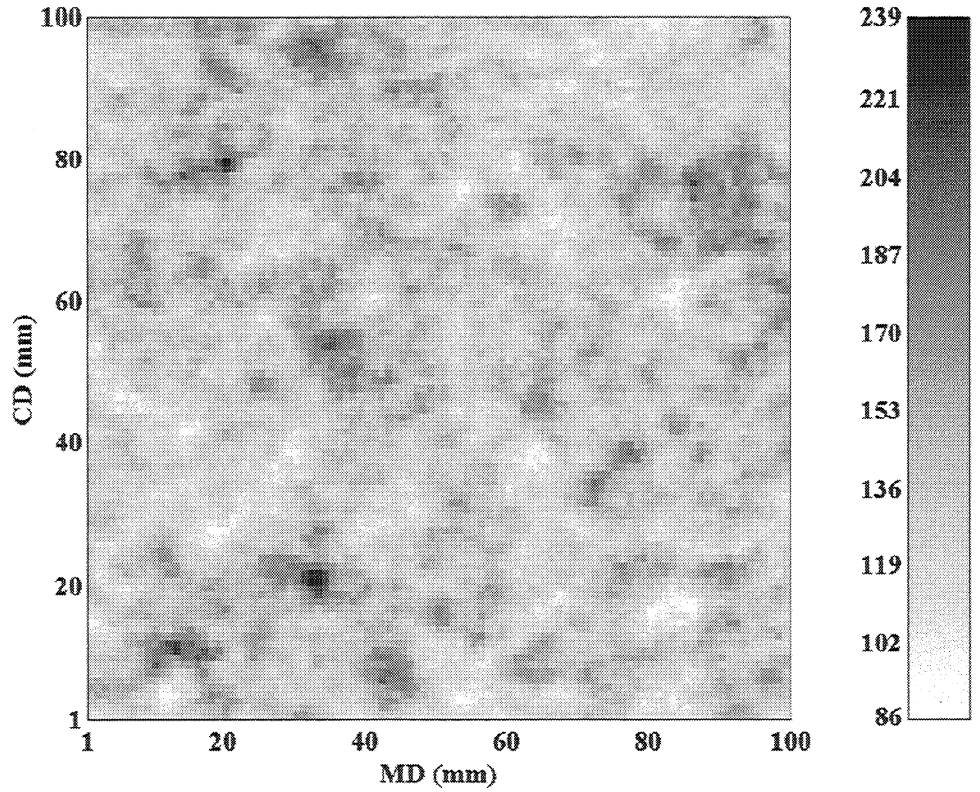

Fig. 4 Sample F grammage [G] map at 1-mm resolution (Z-axis in g/m^2).

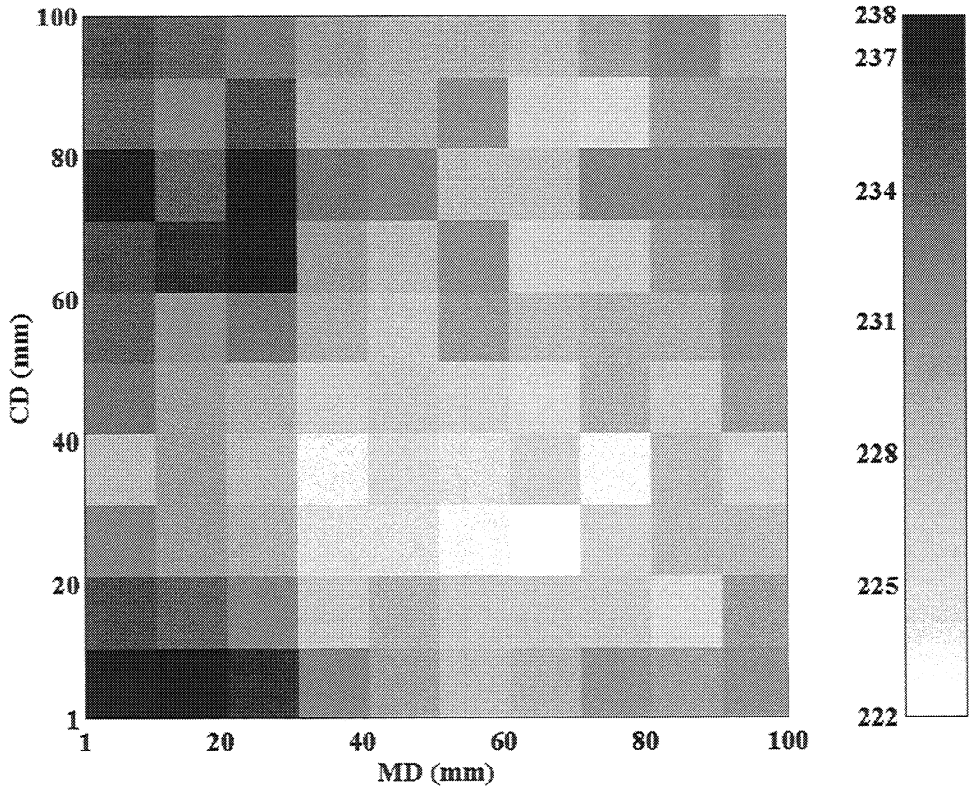

Fig. 5 Sample A grammage [G] map resampled to 10-mm resolution (Z-axis in g/m^2).

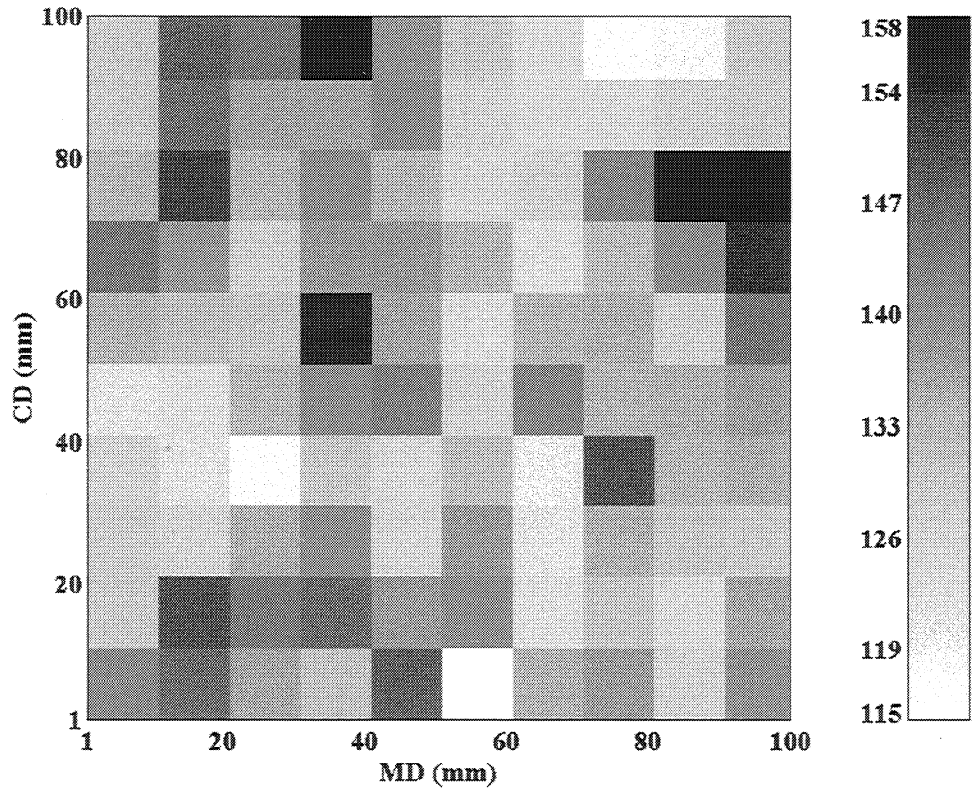

Fig. 6 Sample F grammage [G] map resampled to 10-mm resolution (Z-axis in g/m^2).

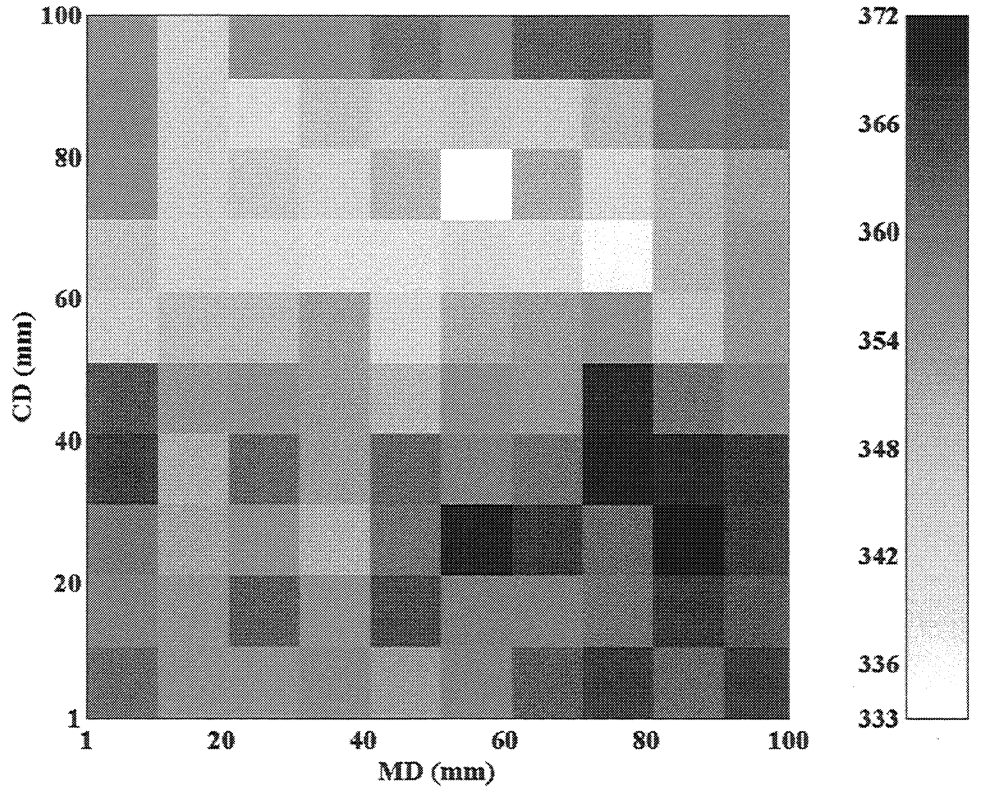

Fig. 7 Sample A soft-platen thickness [h] map at 10-mm resolution (Z-axis in μm).

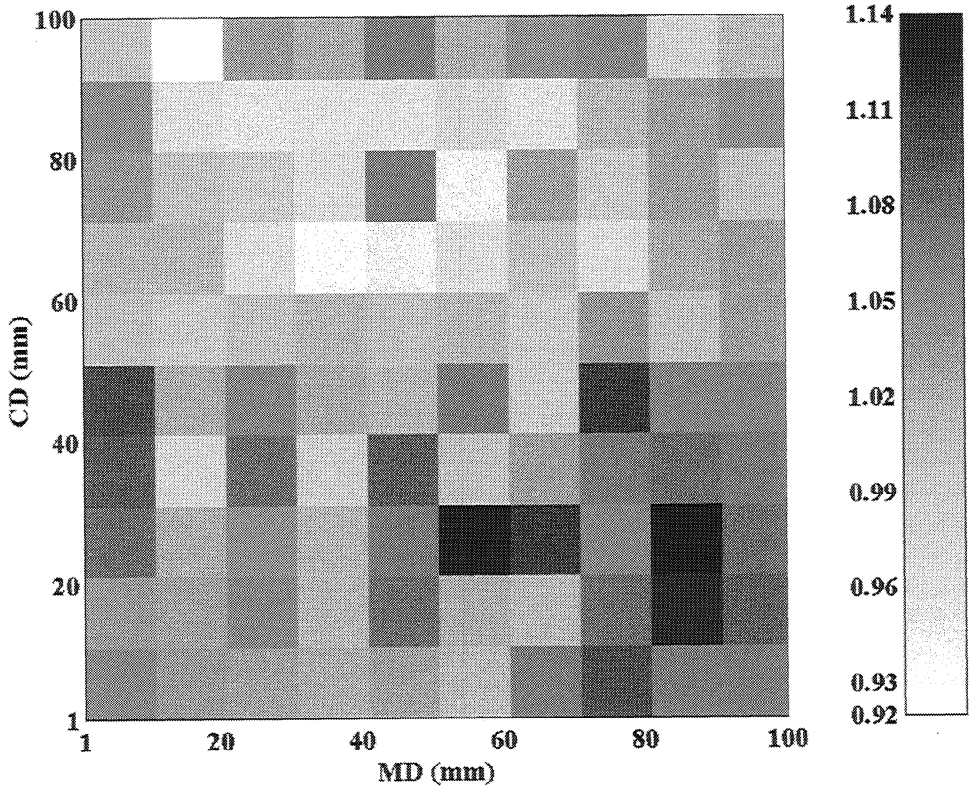

Fig. 8 Sample A traveling time [t] map at 10-mm resolution (Z-axis in μs).

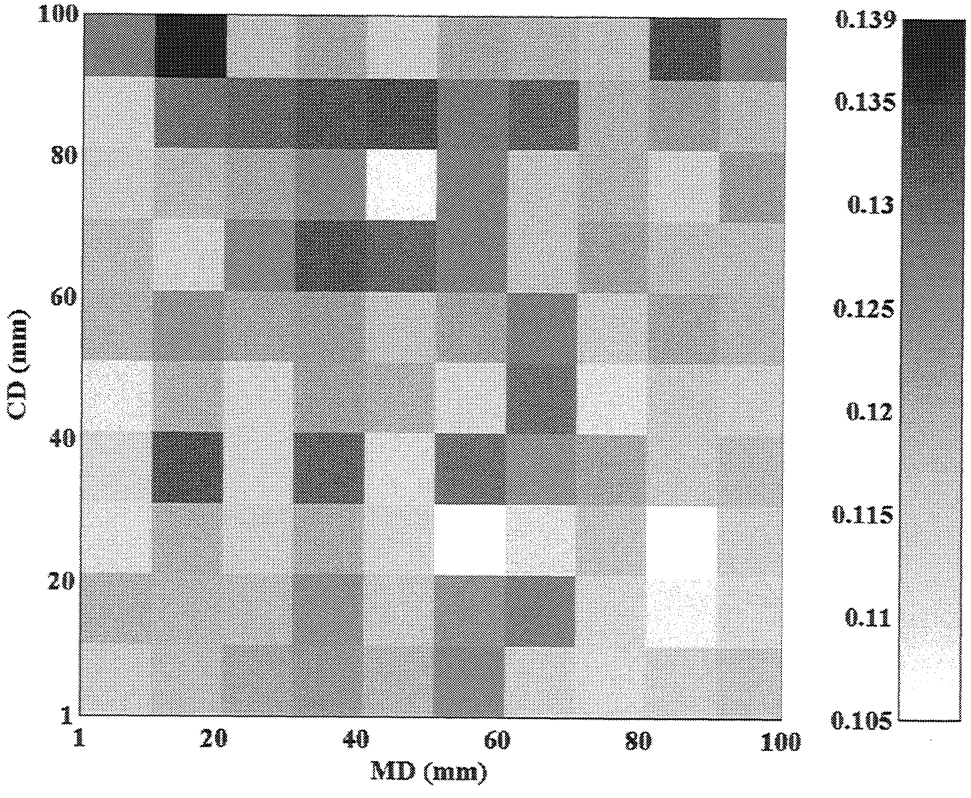

Fig. 9 Sample A specific stiffness [$C_{33}/\rho = (h/t)^2$] map at 10-mm resolution (Z-axis in km^2/sec^2)

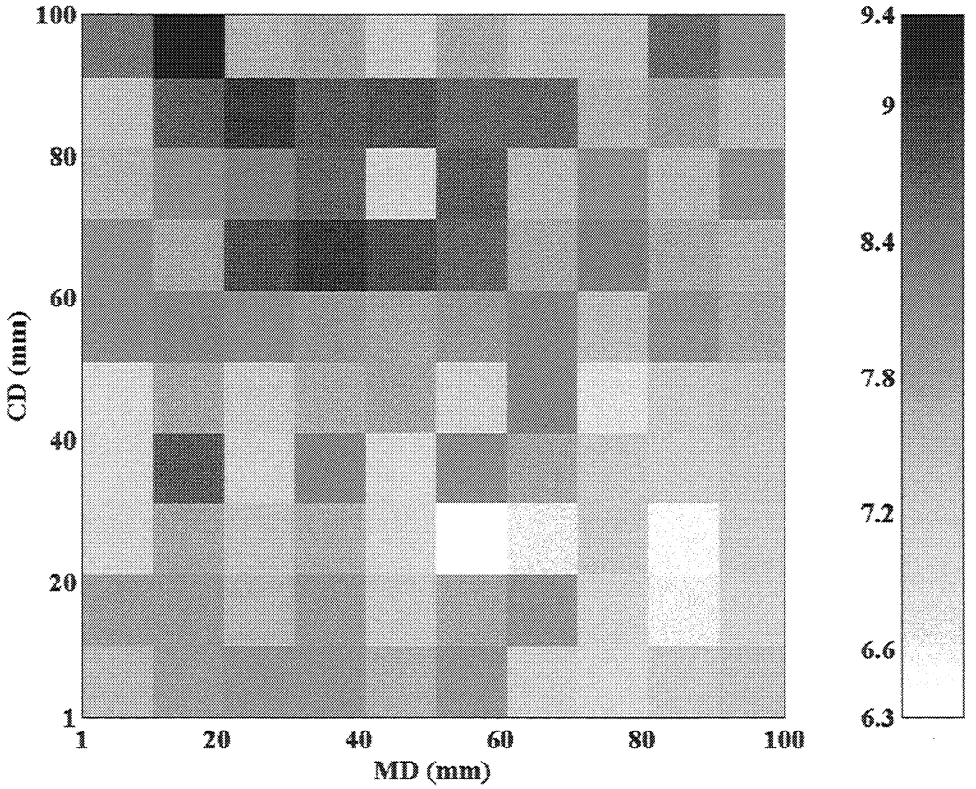

Fig. 10 Sample A elastic stiffness [$C_{33} = \rho v^2 = G h/t^2$] map at 10-mm resolution (Z-axis in MPa).

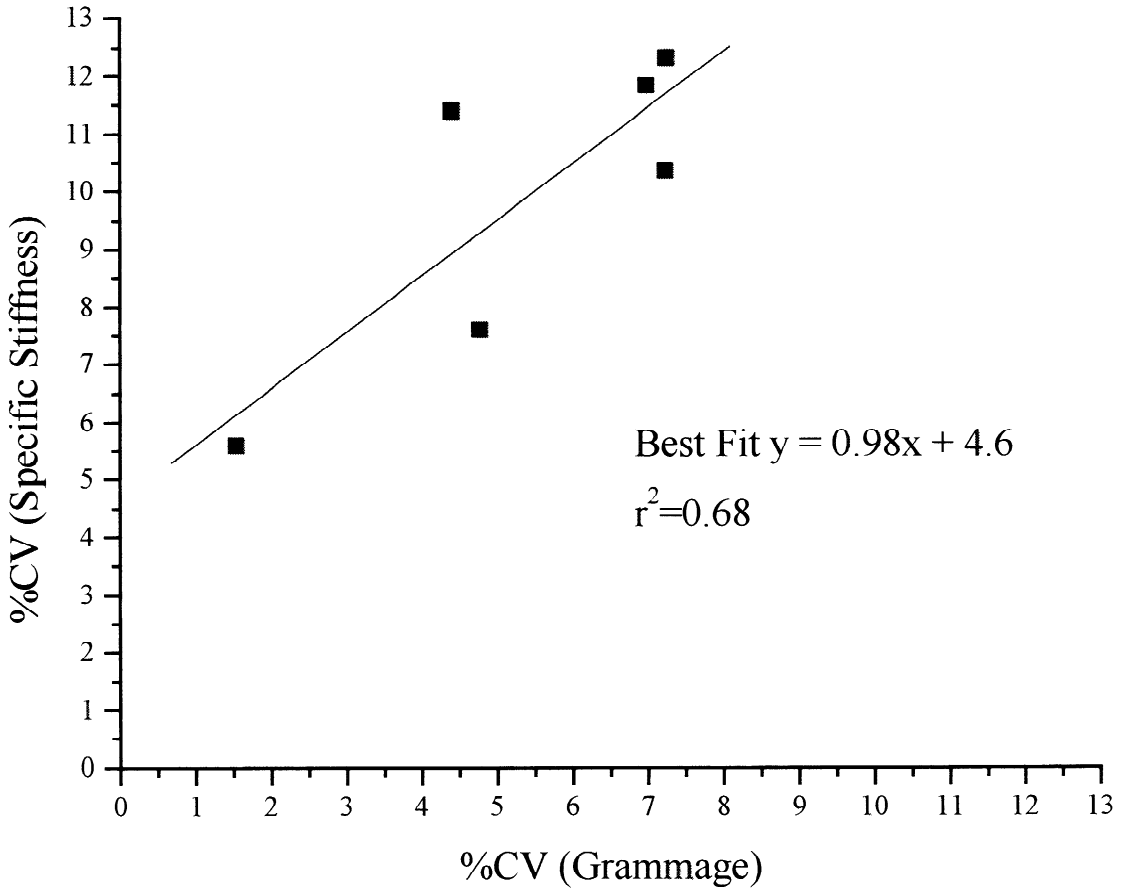

Fig. 11 Coefficient of variation for specific stiffness vs. formation index (%CV for grammage) for all six samples.