The Loss of Some Strength Characteristics of Coated Magazine Paper as a Result of Exposure to Elevated Temperatures

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THE LOSS OF SOME STRENGTH CHARACTERISTICS OF COATED MAGAZINE PAPER AS A RESULT OF EXPOSURE TO ELEVATED TEMPERATURES.

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INTRODUCTION

The purpose of this work is to investigate the effect that varying degrees of temperature have on the strength properties of coated magazine stock. Paper of this type encounters high temperature conditions primarily in the ink drying operations following the printing of the web. The strength with which the paper meets the cutting, folding and binding methods which follow the drying operation are of paramount importance to the printer. Throughout the work, attempts were made to assimilate conditions encountered in present day commercial practice.

LITERATURE SURVEY

COATED MAGAZINE STOCK

Furnish

The furnishes for coated magazine stock vary from mill to mill and depend partly upon the available raw materials and partly upon the desired quality of the paper. The major stocks used are bleached sulphite, bleached hardwood, groundwood, and deinked stock. The structure of a typical coating raw stock contains 25-50 per cent supporting fibres, 40-70 per cent filler fibres, 5-15 per cent filler pigment, 1-10
per cent sizing and other ingredients (1). The cheaper grades of body stock consist solely of sulphite wood mixed with mechanical wood. Time Magazine is an example of coated magazine stock and the body stock is composed of approximately 50 per cent sulphite and 50 per cent groundwood (4). Two typical furnishes representative of this type of paper are either 50 per cent bleached sulphite, 40 per cent groundwood, 10 per cent bleached hardwood or 60 per cent bleached sulphite, 40 per cent soda. Ink drying equipment tends to degrade the strength of the paper and it is necessary to use a relatively high percentage of long fibred chemical pulp in the furnish to keep the web from breaking on the presses even though it is printed at lower speeds than ordinary newsprint the furnish of which usually consists of less than 20 per cent sulphite and over 80 per cent groundwood (4). It is easier to apply heavier weights of coating to the more absorbent sheets such as those with higher groundwood content (2). Sizing of the base sheet is not usually considered to be too important.

Coating Colour

Pigment coating requires the use of two basic raw materials which are the pigment and the adhesive used for bonding the pigment to the paper (1). The common pigments used are clay, calcium carbonate blanc fixe, and satin white while the adhesives employed are starch, casein, soybean protein, synthetic resins, and a small percentage of gums and glues (5). The
greater proportion of magazine coated paper is starch coated because it is inexpensive and its adhesive qualities meet the requirements for this grade of stock.

The coating colour is usually prepared by separately dispersing the pigment and adhesive in water and finally mixing the two together (1). When dry clays are slurried at the mill, they are dispersed in water by means of a dispersing agent, which prevents coagulation of the clay, and the final suspension is brought to approximately 70 per cent solids. The starch is cooked in 34 parts water which results in a cook of about 25 per cent solids. The per cent solids of the final coating colour is in the range of 55-65 per cent and would consist of approximately 84 per cent pigment, 14 per cent adhesive, and 2 per cent plasticizer, wetting agents, softeners, and waxes.

APPLICATION OF COATING COLOUR

Massey Process

Machine coated paper was developed with the primary objective of economically producing a high finish mineral-coated paper with a plane surface capable of perfectly reproducing fine half-tone engravings (6).

The principle of the Massey process is the preparation of a previously determined quantity of coating into a perfect film of the necessary weight and thickness and the simultaneous application of this film to both sides of a web of paper. This is accomplished by rolling pressure without further working
the coating after it is applied to the web. This type of coating process calls for a coating colour of high solids with plasticity predominating over flow property (5).

**Description of the Massey Coater**

Application of the coating to the paper is made on both sides simultaneously by a pair of 48 inch rubber covered rolls mounted one directly above the other. Transfer to the press rolls and preparation of the film is done by a series of transfer rolls extending on a horizontal plane towards the wet end. The problems of crowns is avoided by having all nips in a horizontal plane. Oscillating rolls are provided in each deck to eliminate streaks. The coating is fed to the transfer rolls by a pair of gate rolls in each deck. Pools of colour are maintained in the nips of each pair of gate rolls and fine adjustments of the coat weight are made by regulating the nip pressure of the gate rolls and the speed of rotation. Except for the gate rolls, the linear surface speed of all rolls is approximately the same as that of the paper. The coated sheet, after leaving the press rolls, passes through a short air dryer before going over the remaining dryers. Little moisture is evaporated here but the brief time interval allows the excess moisture in the coating to be absorbed by the sheet so that surface is set and does not stick to the steam dryers. Speeds are limited only by the rate at which satisfactory formation can be obtained on the wire (2).

On sheets below 55 pounds per ream the weight of coating must be decreased to allow a margin in strength factors but
it has proven to be a simple matter to run sheets as light as 40 pounds after coating.

PRINTING AND DRYING OF COATED PAPER

The Printing Process

As the severe drying conditions which are being investigated are almost entirely encountered on typographic or letterpress presses, and as a large proportion of magazine stock is presently printed on web-fed, multi-colour, heat-set presses of this type, our discussion will be limited to letterpress printing.

The relief or letterpress process is the most widely used of the printing processes (1). In relief printing, a raised printing plate surface is covered with ink by rubber or similar type rollers. In the rotary press, the printing plates are fitted around a plate cylinder and the paper web is run between the plate cylinder and the backing or impression cylinder. The ink is then transferred to the surface of the paper under pressure so that part of the ink film transfers to the paper and part stays on the plate. The ideal condition is to transfer the greatest proportion of ink to the paper surface.

In the case of multicolour printing, four basic colours are used - yellow, red, blue, and black (7). Each colour is printed separately with the yellow being laid down first, followed by the red, then the blue, and finally the black. The first ink is the most viscous, and is followed by the
printing of succeeding less viscous inks. The plate is made up of raised areas or dots which lays the ink as a mass of dots on the paper (1). Separate plates are prepared for each of the colours and the desired shades or tones are reproduced by overlapping dots of different coloured inks.

The depth of colour of the impression is determined by the size of the dots and the distance between them. This process is known as halftone reproduction and it is employed in pictorial work. Two other forms of reproduction are used and they are called type reproduction and line reproduction.

Type reproduction is used in the printing of letters, numbers, and characters whereas line reproduction employs full colour values with the ink being laid down as solid lines or areas with no shading.

In multicolour printing, after the necessary colours have been laid down, the paper passes to the dryers. As each surface of the paper is printed separately the use of two dryers is necessary.

Heat-set Printing Inks

The thickest film of ink that can be cleanly printed on the average coated paper is about $12/100,000$ of an inch thick (8). When spread on a hard surface, this would take about fifty hours to dry by natural oxidation which makes it obvious that ink drying by oxidizing of the binder results in well defined time limits. However, the modern trend in printing is towards higher press speeds and present day publication
printing runs in the vicinity of 1,000 feet per minute with some presses reaching speeds of 1,500 feet per minute (7). As a result it is absolutely essential to have almost instantaneous drying of the inks to prevent smearing and smudging in subsequent operations. This problem has been solved by the use of heat-set inks, and high-speed dryers.

Heat-set inks used in relief printing contain a high percentage of volatile solvent of definite evaporation temperature (1). A typical heat-set ink would contain 20 percent pigment, 45 percent resins, plasticizers, waxes, and 35 percent solvent. The solvent is employed to dissolve the resin which acts as the binding agent for the pigment. The solvent is relatively non-volatile at room temperatures but at elevated temperatures is very volatile. When the solvent is evaporated, it leaves the resin to bind the pigment to the paper. The solvent in heat-set inks is a glorified kerosene in that it has been fractionated to yield a liquid of narrow boiling range.

At high speeds it is essential that the ink be highly fluid up to and during the period of impressional contact and almost instantly after become essentially solid (9). For this reason the resins are treated in order to raise the melting point to the vicinity of 300 degrees Fahrenheit (7). When the paper is in the dryer, the resins are actually liquid and the high temperature melting point enables the resin to set quickly when the paper passes over the cooling rolls. An oleoresin-
ous material is also added to the resin (approximately 5 per cent) to make it print smoothly and flow more easily. Flow agents such as candle pitch are included in the ink to help it distribute better on the paper surface.

Most of the drying is done by evaporation although only about 75 per cent of the solvent in the ink is evaporated in the dryers as it would require too much heat to remove all the solvent. Some penetration occurs when the ink is first laid down and the ultimate drying is completed in the next several days by natural oxidation (7).

**Drying the Ink**

The two most common types of dryers used in letterpress printing are drum dryers and flame dryers.

Drum dryers employ steam to attain temperatures of about 350 degrees Fahrenheit. The steam is passed through the hollow drums and the unprinted surface of the sheet is run over the face of the drum. At the same time a blast of hot air plays on the surface of the printed area and helps greatly to dry the ink by evaporating the solvent. The temperature of the hot air is between 500-700 degrees Fahrenheit. These dryers operate at lower temperatures than gas flame dryers and hence do not attack the paper as much (7).

The gas flame dryer directs a jet of flame directly on the printed surface of the web. Other dryers of this type employ radiant heat. The radiant heat units use reflector type heating surfaces which reach surface temperatures up to
1,800 degrees Fahrenheit. This type of drying does not have as severe an effect on the paper as the direct flame dryers because the heat is more diffused although the actual temperature in the oven is often in the range of 1,500-1,800 degrees Fahrenheit (7). As the dryers are from five to seven feet long the paper is subjected to the drying effect only for a period of time of about one-fifth of a second. The intensity of heat application for a given condition is determined by press speed, insulating properties of the paper, moisture content of the paper, and the thickness of the ink layer (8). Usually the surface temperature of the web is kept around 300 degrees Fahrenheit or less (9). However, in present day high-speed heat-set printing, in cases of multi-colour, heavy film jobs, web temperatures of over 400 degrees Fahrenheit may be reached.

When the heated paper leaves the dryer, it is passed over cooling rolls to bring the paper back to room temperature and to complete the setting by making the ink tack-free (1). Some machines apply jets of dry steam to the web immediately after the dryer to help the paper regain moisture (7).

FUNDAMENTALS OF STRENGTH CHARACTERISTICS

Analysis of Strength Factors

Numerous microscopic studies have shown that paper fibres are felted together with all but the shortest surface fibres which lie parallel to the sheet (10). The fibres are all curved, bent, or kinked in a large variety of shapes. It is also known that random orientation is not achieved in machine-
made paper but that the distribution function for direction (relative to the machine direction) displays a maximum in the machine direction and a minimum in the cross-machine direction.

Each fibre lies in contact with, or in near contact with, many other fibres. It is generally believed that relatively strong bonds exist at these points of contact. Undoubtedly the average fibre is bonded to a very large number of other fibres as indicated by the very small effective pore size of most papers (of the order 1/10,000-1/100,000 centimeters). Thus for each fibre stressed during a mechanical test, many fibre to fibre bonds are stressed.

It appears to be generally agreed that fibre to fibre bonds are responsible for the strength of typical papers. Even though these bonds are numerous, failure evidently initiates in them rather than in the fibres. The breaking length of paper (typically in the order of magnitude of 5,000 metres) is much less than that of wood pulp fibres which has been shown by Klauditz, Marschall, and Ginzel (17) to be in the vicinity of 40,000 metres. However, the near random orientation of fibres would account for the lessening of the maximum possible breaking length of paper corresponding to infinite bonding strength.

There are several observations which argue against the theory that the strength of paper resides in the interweaving fibres and fibrils as in a twisted textile thread. One is that paper made from a well beaten pulp is extraordinarily weak
when the water in the wet web on the forming wire is replaced with a non-polar liquid (e.g., when treatment is made with acetone followed by butanol). Another is the fact that certain special pulps, which fibrillate extensively on beating, will not yield strong paper when both the beating and the sheet forming are done in the absence of water. The strongest argument against the mechanical binding theory shows up in the recent work with load-elongation curves. "Primary creep" and "secondary creep" are inherent in normally formed papers. Secondary creep is an irreversible extension which occurs when paper is first strained. Steenberg (18) has shown that a strain-hardened paper (from which secondary creep has been removed) will withstand repeated straining and relaxing without damage to its structure. This behaviour would not be expected if paper bonding was primarily frictional in nature. Furthermore the load-elongation curves obtained by Steenberg and his associates were shown by them to be in accord with the theory of Eyring and Halsey which does not embrace microscopic slipping.

Mason (16) states that the swelling of non-crystalline cellulose by water is the physico-chemical event underlying the whole paper-making process and the mechanical strength of a piece of paper is largely the result of the formation of physico-chemical bonds between water-swollen cellulose surfaces. If water swelling did not occur, paper strength beyond the level of blotting paper could not be produced.
Paper sheets have been made incorporating a small proportion of coloured fibres, and broken edges of such paper have been examined microscopically. It is fairly easy to see which of the coloured fibres lying across the zone of the break have been broken and which have been pulled out from the structure. On papers made from moderately beaten stuff, the proportion broken was about 15 per cent. The other 85 per cent were pulled out, presumably after bond breaking. Only liquids which swell cellulose (such as water) lead to high paper strength.

Mason further suggests that sheet strength may be the resultant of a whole range of types of bonding: there may be the strongest type of bonding resulting from chemical linkage of cellulose chains themselves firmly anchored in the crystalline regions; bonds of the weaker type resulting from the union of unanchored amorphous components; half frictional bonds of a still weaker kind between fibrillae; and the weakest of all, frictional bonds between fibres.

**Folding Endurance of Paper**

Folding endurance is a modification of the tensile strength determination but the flexing ability of the paper affects the results. Casey (1) states that poor folding endurance can be due to short fibre length, poor fibre bonding, fibre brittleness, improper beating, poor formation, and low moisture content. Groundwood papers and those containing hardwood along with those which are heavily filled or coated show low folding endurance. If a mechanical wood pulp is mixed with a sulphite pulp, the
folding endurance of the resulting paper is only slightly better than that of the wood filler pulp.

Van Nederveen and Van Royan (15) found that fibre length clearly affected folding strength as when paper failed in this respect the fibres were not broken but were simply pulled apart. When the fibres were embrittled by treating them with Formaldehyde they snapped upon folding although the tensile strength was practically unaffected.

In his discussion of folding, Mason (16) states that when paper is folded bending stresses occur across the line of the fold and these stresses have a tendency to relax due to the creep in the paper. A paper with a high relaxation rate has a tendency to remain folded whereas resilient papers or those which unfold are those of low relaxation rate. It is believed that papers showing high relaxation rate and high primary creep possess high folding endurance.

Effect of Moisture Content on the Strength of Paper

It is a well known fact that moisture content plays an important part in the physical strength of paper. In a comparison of the strength properties of paper at various per cent relative humidities, the per cent variation of these properties from values at 65 per cent relative humidity was plotted against relative humidities which would result in paper moisture contents of from 0-20. Folding endurance was affected the most of all the common tests as it increased with increasing humidity over the whole range (12). It also
showed the greatest per cent strength loss of all the tests at the lowest paper moisture content. The tearing strength also showed an appreciable loss in strength at low moisture content whereas tensile and bursting strengths were greater at the lowest humidities than they were at 65 per cent relative humidity.

Casey (1) states that the increase in folding endurance with increasing relative humidity is due to an increase in the flexibility of the fibres. Paper should never be over-dried on the paper machine since this results in a more or less permanent loss in strength and flexibility. If paper has been over-dried, conditioning at high humidity (90 per cent) and then bringing back to normal often results in a permanent increase in strength on account of the release of internal stresses set up in the paper during the drying.

Basberg (13) found that the strength of paper reached a maximum at 50 degrees Centigrade and decreased with further heating. At a constant temperature of 20 degrees Centigrade the maximum strength was attained at 1-2 per cent moisture. Paper heated for some time at 100 degrees Centigrade did not recover its original moisture and as the heating temperature grew higher, the less moisture was reabsorbed. The permanent loss of moisture was accompanied by a permanent loss in strength.

**Effect of Dryers on Paper**

The chief disadvantage of heat-set inks is the necessity
of subjecting the paper to high temperature which sometimes causes shrinkage, excessive static electricity, and a dehydration of the paper(1). The strength of the paper is often reduced by as much as 20 per cent during drying. During a series of printing and heat drying test, (web at 250 degrees Fahrenheit), it was revealed that the moisture loss from various papers, starting with initial moistures of 5, 6, and 7 per cent, resulted in final moistures of from 3.2-3.5 per cent. When the travelling web was subjected to heat much in excess of a registered temperature of 250 degrees Fahrenheit at exit of the heating chamber, then the recuperative ability of the interstitial matter was destroyed (8). The theory was advanced that the fibres were partially or totally dehydrated and a chemical transformation occurred that rendered both the interstitial matter and fibres incapable of moisture recovery.

Life Magazine, after printing, is subjected to a temperature of 600 degrees Fahrenheit and the moisture content of the sheet is dropped to about 1 per cent (11). It was estimated that 25 per cent of all web breaks came after the paper passed through the dryers. Tests on the paper before and after going through the press showed that the tensile and mullen strength remained almost the same whereas the tearing strength degraded from 23 to 15 in the machine direction and from 28 to 21 in the cross direction (4).
LITERATURE CITED

(1) Casey J. P., Pulp and Paper, Volume 11
New York, New York, Interscience Publishers, Inc. (1952)

Paper Mill 61, No. 53:15-19 (Dec. 31, 1938)

Oxford University Press (1952)

(4) Thoma P. J., Take the Heat Off, Speaking before the New York
Production Club (1949)

(5) Massey P. J., Paper Machine Coating Processes, Paper Trade J. 109,
No. 16:34-37 (T.S. 210-213) (Oct. 19, 1939)

(T.S. 207-209) (May 24, 1945)

(7) Larsen L., Interchemical Corporation, Personal Interview (Nov. 1953)

(8) Mac Arthur C., Printing With Rapid Drying Inks, Paper Trade J. 15:37
(Oct. 7, 1937)

(9) Engelhart L. F., Printing Ink and Equipment Development and Their
Effect on Paper Requirements, TAPPI 34, No. 8:116-119 (Aug. 1951)

(10) Van Den Akker J., The Elastic and Rheological Properties of Paper


(13) Basberg A., The Influence of Moisture and Temperature on the Strength
and Elasticity of Paper, Paper Journal 21, No. 11:103-105; No. 12:114-117;

(14) Davis J. D., Moisture Content and Regain of Coated Papers, Paper Trade J.
Vol. 113, (July 10, 1941)

(15) Van Nederveen G., and Van Royen A., Some Considerations of the Correlation
Brit. Paper and Board Makers Assoc. 28, No. 10-12:68 (Oct. -Dec. 1951)

(16) Mason S. G., The Rheology of Paper, Pulp and Paper Mag. of Canada
Vol. 49, No. 3 Convention Issue (1948)

(17) Klauditz W., Marschall A., and Ginzel W., The Technology of Lignified
Plant Cell Membranes; Determination of the Tensile Strength of Pulp
Fibres from Pinus Merkusii. Holzforschung I, No. 4:98-103 (1947)

(18) Steenberg, Borge, The Rheology of Paper 1 The Practical Significance
of Paper Rheology, Paper-Maker 118, No. 4:258-259 (Oct. 1949)
EXPERIMENTAL WORK

June 5, 1954
Hugh D. Burnie
EXPERIMENTAL DESIGN

The effect that exposure to elevated temperatures has on coated magazine paper was to be determined by comparing the strength remaining in the paper after heating with the original strength of preconditioned control blanks.

In the tests, a machine coated groundwood - content book paper was used. This sheet designed for commercial publication printing, had a groundwood content of fifty per cent. The remaining stock was bleached sulphite. The paper was of 45 pound basis weight on a 25 X 38 - 500 ream size with an actual sheet size of 17 inches by 22 inches.

All the paper tested was preconditioned at fifty per cent relative humidity and seventy-three degrees Fahrenheit for forty-eight hours previous to testing.

To secure a sufficient number of tests, three separate sheets were tested at each temperature and exposure period. In the case of the control blanks five separate sheets were tested. The basis weight of each sample was determined following the forty-eight hour pre-conditioning period. In order to facilitate handling of the samples in the ovens, the test sheets were cut into four equal strips across the grain. This was done after the paper had been preconditioned for the forty-eight hour period.

The ovens were raised to the desired temperature and the test strips were placed in them in such a manner as to allow free circulation of the hot air around the entire sample.
This was accomplished by the use of wire supports set up in the ovens. In cases where the exposure period was short, the required temperature was exceeded by a slight margin to allow for the drop in temperature which was caused by the opening of the oven door when the samples were being introduced.

At the end of the exposure period, the test strip was removed from the oven and immediately placed in a desiccator to allow it to cool before testing. The sample was desiccated for a period not exceeding five minutes. After desiccation, the test strips were cut into the necessary sizes for the various tests. The tests carried out on the sheets following the exposure period were the interval tear test, the tensile test, and the folding endurance test.

Sufficient tests were carried out on each sample so that the total number of determinations of each strength property, in both the machine direction and cross direction, lay between fifteen and twenty for each test condition. The following outline indicates the proposed experimental work, showing the exposure periods and temperatures.
<table>
<thead>
<tr>
<th>Designation</th>
<th>Exposure Period</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample A</td>
<td></td>
<td></td>
</tr>
<tr>
<td>&quot; B-1</td>
<td>48 hours</td>
<td>73 deg. F. &amp; 50% R.H.</td>
</tr>
<tr>
<td>&quot; B-2</td>
<td>15 minutes</td>
<td>105 deg. C. (230 deg. F.)</td>
</tr>
<tr>
<td>&quot; B-3</td>
<td>1 hour</td>
<td>105 deg. C. (230 deg. F.)</td>
</tr>
<tr>
<td>&quot; C-1</td>
<td>24 hours</td>
<td>105 deg. C. (230 deg. F.)</td>
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<tr>
<td>&quot; C-2</td>
<td>1 minute</td>
<td>140 deg. C. (284 deg. F.)</td>
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<tr>
<td>&quot; C-3</td>
<td>15 minutes</td>
<td>140 deg. C. (284 deg. F.)</td>
</tr>
<tr>
<td>&quot; D-1</td>
<td>1 hour</td>
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<td>&quot; D-2</td>
<td>15 seconds</td>
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<tr>
<td>&quot; E-1</td>
<td>1 minute</td>
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<tr>
<td>&quot; E-2</td>
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<tr>
<td>&quot; F-1</td>
<td>5 seconds</td>
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</tr>
<tr>
<td></td>
<td>1 second</td>
<td>560 deg. C. (1076 deg. F.)</td>
</tr>
</tbody>
</table>
EXPERIMENTAL WORK

Description of Test Paper

In carrying out the experimental work, the necessity of running the tests on a regular commercial grade of paper having an appreciable groundwood content was recognized as being a prime importance. The paper which was tested, fulfilled these requirements in that it consisted of fifty per cent groundwood stock along with fifty per cent bleached sulphite. The stock was selected from a normal run of one-machine two-side coated magazine paper of forty-five pound basis weight on a 25 X 38 - 500 ream size. The raw stock sheet was thirty-three pounds with a six pound coat on each surface. The coating consisted of an enzyme converted starch adhesive with clay as the pigment. The actual sheet size as delivered was seventeen inches by twenty-two inches, with the grain in the long direction.

Evaluation of Control Blanks

In order to secure accurate control data which would indicate the strength of the paper before it was subjected to above normal temperatures, it was decided to run tests on five sheets (17 X 22) so that at least twenty-five tests would be obtained for each strength characteristic in both the machine direction and the cross direction. It was felt that this large number of tests was necessary to eliminate any doubtful variations which would occur in test values. The sheets were hung for forty-eight hours in an automatically controlled humidity room in which the relative humidity was maintained
at fifty per cent and the temperature was stabalized at seventy-three degrees Fahrenheit. Following this forty-eight hour preconditioning period, a basis weight test was run on each sheet according to Tappi Standard T 410 m - 45. This was done so that corrections could be carried out on the tests to allow for variations in sheet weight. The samples were then tested for internal tear strength, tensil strength, and folding endurance.

Testing Instruments and Procedures.

Internal Tear Strength

The internal tear strength tests were carried out on a conventional Elmendorf Tear Tester using the standard Tappi procedure T 414 m - 49. In all cases, eight sheets were torn at one time and the values were doubled and recorded for sixteen sheets.

Tensile Strength

The tensile strength evaluations were run on a Schopper Type Tensile Tester which was motor driven. The jaws of the tester were set two inches apart at the start of each test and each sample was cut so that it was fifteen millimetres wide. In other respects, the tests were run as suggested in Tappi Standard T 404 m - 50.

Folding Endurance

A Massachusetts Institute of Technology Folding Tester was employed for the folding endurance tests. Tappi Standard T 423 m - 50, Section 11 was followed throughout the tests with 0.5 Kilograms tension applied to the paper.
General Testing Procedures

Conditioning and Preparing Specimens

Each sheet of paper tested was first conditioned for forty-eight hours at seventy-three degrees Fahrenheit and fifty per cent relative humidity. The sheets, which were seventeen inches by twenty-two inches, were suspended at two points on a conditioning rack which allowed for free circulation of the air around the samples. A basis weight test was then carried out on the sheet according to Tappi standards. The sheets were then cut in a cross grain direction into four equal strips and these samples were placed in the ovens.

Testing Ovens and Heating Procedures

In the lower temperature range of from 105 degrees Centigrade to 180 degrees Centigrade, the samples were heated in a Precision Scientific Co. oven. This oven is thermostatically regulated and is normally used for laboratory moisture tests. The actual temperature in the oven is read on a thermometer which is mounted in the top of the oven so that the mercury bulb projects directly into the air in the upper portion of the oven box. Entry into the oven is made by means of a door two and one-half feet high by two feet wide.

Initial attempts at heating the samples indicated that they could not simply be placed on the oven floor as they grouped together and occasionally fell over with the result that the heating effect on the strips was not of a uniform nature. To correct this condition, it was realized that
freer circulation of the air around the samples was necessary. As a result, a series of wires was strung across the oven, six inches from the roof. Each sample strip was hung over a wire and in this way the specimens only contacted metal where they actually touched the supporting wire.

As a certain amount of time was required to place the sample strips in position in the oven, a drop in oven temperature occurred. To remedy this situation, it was found necessary to bring the oven temperature to a slightly higher point than the actual desired heating value. This precaution was not found necessary when the exposure period exceeded fifteen minutes.

In the higher temperature range of from two hundred and sixty degrees Centigrade to five hundred and eighty degrees Centigrade, the samples were heated in a Modern Electric Laboratory muffle furnace. This furnace was thermostatically regulated and was capable of reaching a temperature of eleven hundred degrees Centigrade. The oven temperature is read on a meter which is connected with the furnace pyrometer.

It was found that the sample strips as previously cut in the humidity room, were too large to be easily placed in the furnace. As a result the strips were cut to smaller sizes which more readily fitted in the furnace. The strips could not be placed on the oven floor as no provision was made in the oven for free circulation of the air. This resulted in non-uniform heating. To secure improved heating effects, a wire frame was fabricated. This frame rested on four supporting legs and was so designed that the paper hung between these
legs at the mid point of the oven. The strips were held at only two points and these were located at the ends of the samples.

Desiccation and Testing of Samples

Upon the completion of the heat exposure period, the paper strips were removed from the ovens and immediately placed in a desiccator. The samples were allowed to cool in the desiccator for a period not exceeding five minutes, following which they were tested for their strength properties. To further prevent moisture pick-up by the sample during the testing period, each test was run on only one sample at a time which reduced the exposure period of the strips to the air.

All tests were run in the manner as described for the control blanks. Three individual sheets were tested for each temperature-exposure condition and five tests were carried out on each strength property in each sheet. As a result, a total of fifteen values was obtained on each test.

Fabulation of Results

The averages of the fifteen tests for each set of conditions are listed in Table I under the heading 'Average of Individual Test Results'. A further breakdown of the results is listed in Table III under the heading 'Summary of Test Results'. Graphical representation of the results are shown in figures 1, 2, and 3.

Discussion of the Results

(a) Folding Endurance.

The folding endurance of the paper was very seriously decreased by exposure to heat and this effect
### Averages of Individual Test Results

<table>
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<tr>
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<th>Exposure Temperature - degrees Centigrade</th>
<th>Exposure Time - as indicated</th>
</tr>
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<tbody>
<tr>
<td></td>
<td></td>
<td>23</td>
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<tr>
<td></td>
<td>Temp.</td>
<td>48 hrs.</td>
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<tr>
<td>Test</td>
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<td>M.D.</td>
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<td></td>
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<td>C.D.</td>
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<td></td>
<td>M.D.</td>
</tr>
<tr>
<td></td>
<td>Tensile - lbs. per 15 mm's.</td>
<td>C.D.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Tear</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Basis Weight</td>
</tr>
</tbody>
</table>

|                      | Temp.                                      | 180 | 180 | 260 | 260 | 580 |
|                      | Time                                        | 15 sec. | 1 min. | 1 sec. | 5 sec. | 1 sec. |
| Test                 |                                             | M.D. | 468 | 433 | 718 | 30  | 3   |
|                      | Fold - M.I.T. Double Folds                 | C.D. | 121 | 79  | 122 | 11  | -   |
|                      |                                             | M.D. | 10.10 | 9.64 | 9.09 | 9.34 | 4.17 |
|                      | Tensile - lbs per 15 mm:                   | C.D. | 4.68 | 4.68 | 4.60 | 3.95 | 1.95 |
|                      |                                             | Tear | 17.3 | 16.7 | 17.9 | 12.7 | 9.3 |
|                      |                                             | Basis Weight | 43.9 | 44.0 | 44.3 | 44.0 | 44.5 |
Averages of Individual Test Results  
Corrected for Variations in basis weight.

Exposure Temperature - degrees Centigrade  
Exposure Time - As indicated

<table>
<thead>
<tr>
<th>TABLE II</th>
<th>Temp. 23</th>
<th>105</th>
<th>105</th>
<th>105</th>
<th>140</th>
<th>140</th>
<th>140</th>
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</thead>
<tbody>
<tr>
<td>Time</td>
<td>48 hrs.</td>
<td>15 mins.</td>
<td>1 hr.</td>
<td>24 hr.</td>
<td>1 min.</td>
<td>15 min.</td>
<td>1 hr.</td>
</tr>
<tr>
<td>Test</td>
<td>Fold - M.I.T. Double Folds</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>M.D.</td>
<td>886</td>
<td>713</td>
<td>527</td>
<td>476</td>
<td>565</td>
<td>276</td>
</tr>
<tr>
<td></td>
<td>C.D.</td>
<td>131</td>
<td>147</td>
<td>84</td>
<td>82</td>
<td>73</td>
<td>52</td>
</tr>
<tr>
<td>Tensile - Lbs. per 15 mm strip</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>M.D.</td>
<td>10.1</td>
<td>10.3</td>
<td>9.7</td>
<td>9.7</td>
<td>9.8</td>
<td>10.2</td>
</tr>
<tr>
<td></td>
<td>C.D.</td>
<td>4.4</td>
<td>4.9</td>
<td>4.7</td>
<td>4.7</td>
<td>4.5</td>
<td>4.6</td>
</tr>
<tr>
<td>Tear - Grams</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>M.D.</td>
<td>17.7</td>
<td>12.6</td>
<td>12.6</td>
<td>12.7</td>
<td>14.2</td>
<td>12.0</td>
</tr>
<tr>
<td></td>
<td>C.D.</td>
<td>23.2</td>
<td>17.2</td>
<td>17.4</td>
<td>16.9</td>
<td>18.7</td>
<td>16.6</td>
</tr>
<tr>
<td>Basis Weight</td>
<td>45.0</td>
<td>45.0</td>
<td>45.0</td>
<td>45.0</td>
<td>45.0</td>
<td>45.0</td>
<td>45.0</td>
</tr>
</tbody>
</table>

| Temp. 180 | 180 | 260 | 260 | 580 |
| Time | 15 sec. | 1 min. | 1 sec. | 5 sec. | 1 sec. |
| Test | Fold - M.I.T. Double Folds | | | | | | |
|          | M.D. | 480 | 442 | 729 | 31  | 3  |
|          | C.D. | 124 | 81  | 124 | 11  | - |
| Tensile - lbs per 15 mm strip | | | | | | | |
|          | M.D. | 10.3 | 9.3  | 9.2 | 8.5 | 4.2 |
|          | C.D. | 4.3  | 4.3  | 4.7 | 4.0 | 2.0 |
| Tear - Grams | | | | | | | |
|          | M.D. | 13.0 | 12.4 | 13.8 | 8.9 | 4.7 |
|          | C.D. | 17.7 | 17.1 | 18.2 | 13.0 | 9.4 |
| Basis Weight | 45.0 | 45.0 | 45.0 | 45.0 | 45.0 | 45.0 | 45.0 |
## Summary of Test Results

**TABLE III**  

<table>
<thead>
<tr>
<th>Exposure Temperature - degrees Centigrade</th>
<th>Exposure Time - as indicated</th>
</tr>
</thead>
<tbody>
<tr>
<td>M.I.T. Fold M. D.</td>
<td>Time 0 sec. 1 sec. 5 sec. 15 sec. 1 min. 15 min. 1 hr. 24 hr.</td>
</tr>
<tr>
<td>Temp.</td>
<td>105 140 180 260 580</td>
</tr>
<tr>
<td></td>
<td>886 886 886 728 886</td>
</tr>
<tr>
<td></td>
<td>713 565 480 51 3</td>
</tr>
<tr>
<td>M.I.T. Fold C. D.</td>
<td>Time 0 sec. 1 sec. 5 sec. 15 sec. 1 min. 15 min. 1 hr. 24 hr.</td>
</tr>
<tr>
<td>Temp.</td>
<td>105 140 180 260 580</td>
</tr>
<tr>
<td></td>
<td>131 131 131 124 131</td>
</tr>
<tr>
<td></td>
<td>147 73 124 11</td>
</tr>
<tr>
<td>M.I.T. Tensile - M. D.</td>
<td>Time 0 sec. 1 sec. 5 sec. 15 sec. 1 min. 15 min. 1 hr. 24 hr.</td>
</tr>
<tr>
<td>Temp.</td>
<td>105 140 180 260 580</td>
</tr>
<tr>
<td></td>
<td>10.1 10.1 10.1 10.1 10.1</td>
</tr>
<tr>
<td></td>
<td>9.9 9.9 9.9</td>
</tr>
<tr>
<td>M.I.T. Tensile - C. D.</td>
<td>Time 0 sec. 1 sec. 5 sec. 15 sec. 1 min. 15 min. 1 hr. 24 hr.</td>
</tr>
<tr>
<td>Temp.</td>
<td>105 140 180 260 580</td>
</tr>
<tr>
<td></td>
<td>4.4 4.4 4.4 4.4 4.4</td>
</tr>
<tr>
<td></td>
<td>4.9 4.5 4.8 4.0</td>
</tr>
<tr>
<td>M.I.T. Tear M. D.</td>
<td>Time 0 sec. 1 sec. 5 sec. 15 sec. 1 min. 15 min. 1 hr. 24 hr.</td>
</tr>
<tr>
<td>Temp.</td>
<td>105 140 180 260 580</td>
</tr>
<tr>
<td></td>
<td>17.7 17.7 17.7 17.7 17.7</td>
</tr>
<tr>
<td></td>
<td>12.6 14.2 13.0 13.3</td>
</tr>
</tbody>
</table>

- **M.I.T. Fold M. D.**  
  - Temperature: 105°C, 140°C, 180°C, 260°C, 580°C  
  - Time: 0 sec., 1 sec., 5 sec., 15 sec., 1 min., 15 min., 1 hr., 24 hr.  
  - Values for each temperature and time period are listed.  

- **M.I.T. Fold C. D.**  
  - Temperature: 105°C, 140°C, 180°C, 260°C, 580°C  
  - Time: 0 sec., 1 sec., 5 sec., 15 sec., 1 min., 15 min., 1 hr., 24 hr.  
  - Values for each temperature and time period are listed.  

- **M.I.T. Tensile - M. D.**  
  - Temperature: 105°C, 140°C, 180°C, 260°C, 580°C  
  - Time: 0 sec., 1 sec., 5 sec., 15 sec., 1 min., 15 min., 1 hr., 24 hr.  
  - Values for each temperature and time period are listed.  

- **M.I.T. Tensile - C. D.**  
  - Temperature: 105°C, 140°C, 180°C, 260°C, 580°C  
  - Time: 0 sec., 1 sec., 5 sec., 15 sec., 1 min., 15 min., 1 hr., 24 hr.  
  - Values for each temperature and time period are listed.  

- **M.I.T. Tear M. D.**  
  - Temperature: 105°C, 140°C, 180°C, 260°C, 580°C  
  - Time: 0 sec., 1 sec., 5 sec., 15 sec., 1 min., 15 min., 1 hr., 24 hr.  
  - Values for each temperature and time period are listed.
<table>
<thead>
<tr>
<th>Temp (°F)</th>
<th>0 sec</th>
<th>1 sec</th>
<th>5 sec</th>
<th>15 sec</th>
<th>1 min</th>
<th>15 min</th>
<th>1 hr</th>
<th>24 hr</th>
</tr>
</thead>
<tbody>
<tr>
<td>105</td>
<td>23.2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>17.2</td>
<td>17.4</td>
<td>16.9</td>
</tr>
<tr>
<td>140</td>
<td>23.2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>18.7</td>
<td>16.6</td>
<td>16.5</td>
</tr>
<tr>
<td>180</td>
<td>23.2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>17.7</td>
<td>17.1</td>
<td></td>
</tr>
<tr>
<td>260</td>
<td>23.2</td>
<td>13.2</td>
<td>13.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>580</td>
<td>23.2</td>
<td>9.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
THE EFFECT OF ELEVATED TEMPERATURES ON THE INTERNAL TEARING RESISTANCE FOR VARIOUS EXPOSURE PERIODS

**Figure 1**

<table>
<thead>
<tr>
<th>Control</th>
<th>105 °C</th>
<th>140 °C</th>
<th>180 °C</th>
<th>260 °C</th>
<th>580 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>15 min</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1 hour</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2 hours</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**MACHINE DIRECTION - grams**

**INTERNAL TEARING - grams**

**CROSS DIRECTION - grams**
THE EFFECT OF ELEVATED TEMPERATURES ON FOLDING ENDURANCE FOR VARIOUS EXPOSURE PERIODS

Control 105° Cent. 140° Cent. 180° Cent. 260° Cent. 580° Cent

15 min. 1 hour 24 hour 1 min. 15 min. 1 hour 15 sec. 1 min. 1 sec. 5 sec. 1 sec

Figure 2
THE EFFECT OF ELEVATED TEMPERATURES ON TENSILE STRENGTH FOR VARIOUS EXPOSURE PERIODS

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Exposure Period</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td></td>
</tr>
<tr>
<td>105°C</td>
<td>15 min.</td>
</tr>
<tr>
<td>140°C</td>
<td>1 hr.</td>
</tr>
<tr>
<td>180°C</td>
<td>15 min.</td>
</tr>
<tr>
<td>260°C</td>
<td>1 sec.</td>
</tr>
<tr>
<td>580°C</td>
<td>1 sec.</td>
</tr>
</tbody>
</table>

FiguRe 3
became more pronounced as the temperatures was raised and
the exposure periods lengthened. At 260 degrees Centigrade,
the effect that the initial evaporation of the moisture in
the paper had on the strength became strikingly evident.
After one second exposure at this temperature, the machine
direction fold strength dropped eighteen per cent, whereas
after five seconds exposure the fold strength had decreased
ninety-six per cent. Apparently, in the one second heating
period, the sample was only exposed to the heat long enough
to drive off the moisture in the paper. The strength drop
resulted simply from the drying out of the sample. In the
five second heating period, the paper moisture was flashed
off in the first second or so and in the remaining time the
heat attacked the cellulose and caused rapid degradation in
paper strength.

When the exposure temperature was raised to 580 degrees
Centigrade, the heat was so intense that instantaneous
evaporation of the moisture occurred. As a result, almost
complete degradation of the paper was brought about even
though the exposure period was but one second. This
temperature is well above the combustion temperature of paper
and it was only because of the short exposure period coupled
with the cooling effect in the moisture evaporation period
that the paper did not ignite. The extent of the attack on
the cellulose was indicated by the fact that no cross direction
fold strength figures could be obtained due to the extreme
brittleness of the paper. It required but one flexing of
the specimen to cause it to break when it was in this condition.
(b) Tensile Strength

Tensile strength was the least seriously affected of the strength properties tested. In nearly all instances, when the heating was carried out below 260 degrees Centigrade the resulting tensile strength actually showed an increase of up to two per cent as compared to the control blank values. The first appreciable sign of strength loss occurred after five seconds exposure at 260 degrees Centigrade. In this instance, the strength was reduced fifteen percent in the machine direction and ten per cent in the cross direction. Serious reduction in strength took place when the paper was exposed for one second at 580 degrees Centigrade. The machine direction tensile strength dropped from 10.1 pounds per 15 millimetres of width to 4.2 pounds which was a loss of 58 per cent. At the same time, the cross direction tensile strength was reduced from 4.4 pounds per 15 millimetres of width to 2.0 pounds. This was a 54 per cent decrease in strength. The effect on the strength at this temperature appeared to be another case in which the loss in strength was actually due to partial destruction of the cellulose other than simple loss due to drying out of the sample.

(c) Internal Tear ing Resistance

The internal tearing resistance showed noticeable loss under all the conditions tested and very serious strength losses occurred in some instances. At a temperature of 105 degrees Centigrade, the maximum strength loss appeared to have been reached in an exposure time of fifteen minutes or less and continued heating at this temperature caused no further
reduction in strength. The machine direction tear strength was reduced from 17.7 grams per sheet to 12.6 grams per sheet at this temperature. The cross direction tear strength decreased from 23.2 grams per sheet to an average of 17.2 grams per sheet. Under all the test conditions up to and including 180 degrees Centigrade for one minute exposure, a fairly uniform reduction in tear strength appeared to take place. The machine direction values reached a low of 11.9 grams per sheet at 140 degrees Centigrade with one hour exposure time and the majority of the other strength figures were between 12 and 13 grams per sheet. A similar condition existed in the cross direction strength values as a low of 16.5 grams per sheet was reached after one hour exposure at 140 degrees Centigrade. The majority of the other cross machine test results were between 16.5 and 17.5 grams per sheet. Thus a drop of approximately thirty per cent occurred in both machine and cross direction strengths.

The first break from this range took place after five seconds exposure at 260 degrees Centigrade. The machine direction tear dropped to 8.9 grams per sheet and the cross direction tear dropped to 13.0 grams per sheet. This was a loss of about 52 per cent in tearing resistance in both directions. As in the case of the fold and tensile strength figures, the greatest reduction in tear strength occurred after one second exposure at 580 degrees Centigrade. The strength of the samples was dropped approximately 65 per cent under these conditions.

Generally the tear strength approached fairly constant figures in the lower temperature test range up to and including
180 degrees Centigrade. It is probable that the loss of strength up to this point was simply a result of the evaporation of the moisture in the paper. Beyond this temperature range, the effect of the moisture loss on the strength was augmented by the actual physical decomposition of the cellulose by the heat.

CONCLUSIONS

All the tests results indicate that the folding endurance, the internal tearing resistance, and the tensile strength are reduced to some extent by exposure to temperatures in the magnitude of 260 degrees Centigrade. At temperatures below this value, the tensile strength appears to be relatively unaffected whereas the folding endurance and tear strength show definite weakening. This weakening effect in tear and fold reaches a maximum and further exposure in this lower temperature range has no further effect on the strength of the paper. Apparently the decrease in strength up to this point is due entirely to the evaporation of the moisture in the paper by the heat and no actual attack on the cellulose has occurred.

At 260 degrees Centigrade and higher, the length of the exposure period assumes greater importance. If the paper is heated at these elevated temperatures for a very short time, the only loss in strength is due to loss in moisture content. However, if the heating period is extended until all the water in the paper is evaporated, the cellulose is actually decomposed with the result that severe degradation in strength values occurs.

The folding endurance is the most seriously affected
of the strength characteristics throughout the entire heating range. It is possible to practically remove all the folding endurance of the paper even though some tearing and tensile strength remain in the sheet.