EFFECT OF GAMMA IRRADIATION ON THE TENSILE CHARACTERISTICS OF VISCOSE AND HIGH WET MODULUS RAYON

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Viscose and high wet modulus rayon staple fibres were irradiated uncovered in air with gamma rays in a Gammacell 220. Dosages ranged from 0 rads to 3.0×10^7 rads at a dose rate of 1.21×10^6 rads/hour. Breaking strength, work to break and initial modulus were measured on 25 single fibres at each treatment level. All measurements followed ASTM methods.

Both breaking tenacity and work to break decreased with increasing irradiation. The slopes of the regression lines of these properties and of their logarithms on the irradiation dosages indicated that the high wet modulus rayon was more rapidly degraded. This was attributed to the larger size of its crystalline areas. With both rayons the work to break appeared to be affected more than the tenacity.

The initial modulus of the viscose rayon increased with increasing irradiation. No definite relationship could be established between the initial modulus of the high wet modulus rayon and the irradiation dosage.

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INTRODUCTION

Gamma radiation has been used successfully in the following textile applications:

1. initiating graft polymerization of monomers and polymers on to textile polymers (33);
2. measuring the density of spinning and finishing solutions through tanks and pipes (33);
3. detecting and identifying minute amounts of contaminants in finishes (33); and
4. sterilizing medical supplies such as cotton sutures (9).

Thus information on the effect of gamma radiation on the mechanical properties of textile fibres is important and of particular interest.

Studies have reported the effects of gamma radiation on cellulose in the form of cotton and viscose rayon but little information is available concerning its effects on modified and polynosic rayons.

The development of these modified rayons is an attempt to produce a man-made cellulose fibre possessing some of the desirable properties of cotton that viscose rayon lacks. Modified rayons have improved dry and wet strengths, better resistance to caustic alkali and shrinkage, as well as a higher modulus both wet and dry. These characteristics have contributed to promising acceptance of modified rayons as a substitute for cotton in many blended fabrics (23).

With the extensive use of gamma irradiation of textile polymers, and the present and potential market for modified rayons, the effects of gamma rays on modified rayons are worth investigating. Consequently the purposes of this study were to:

1. determine the tensile characteristics of high wet modulus rayon staple fibres irradiated in air with gamma rays; and
2. compare the tensile characteristics of high wet modulus rayon with those of viscose rayon following gamma irradiation in air.

REVIEW OF LITERATURE

In recent years, cellulose in the form of wood cellulose, cotton and viscose rayon exposed to various high-energy radiations has been the subject of a number of studies.

Many of the researchers exposed cellulose to gamma rays, the radiations of the electromagnetic spectrum with the shortest wavelengths and the highest energies. Gamma rays can transfer their energy to matter by three processes: 1) ionization, 2) excitation and 3) the formation of free radicals. Textile polymers exposed to gamma radiation undergo cross-linking or degradation. In many polymers, both occur simultaneously with one predominating. Textile fibres subjected to other forms of high-energy radiation such as alpha rays, thermal neutrons, cathode rays, beta rays and X-rays in various atmospheres are affected in a similar manner, provided the total dosage received is the same (5, 38). Most of these investigations were carried out in an atmosphere of air, but even those done *in vacuo* or in nitrogen resulted in similar chemical and physical changes (4, 6, 25). Generally, the presence of oxygen in the atmosphere slightly enhanced the reaction (4, 6).

Despite differences in the experimental conditions, investigators have observed the following chemical changes in irradiated cellulose: depolymerization, oxidation and free radical formation.

Saeman *et al.*(35) irradiated wood pulp and cotton linters in air with cathode rays. They observed decreased viscosity in cellulose solvents, indicating depolymerization. This result has been confirmed by other workers (6, 7, 8, 21, 22, 30, 31, 38) using both gamma rays and thermal neutrons. Another indication of depolymerization, increased solubility in water and dilute alkali, was noted by Saeman *et al.*(35), Gilfillan and Linden (20) and Blouin and co-workers (4, 6), using various high-energy radiations.

The total irradiation dosage received and not the dose rate determines the extent of depolymerization (5, 38). A linear relationship between viscosity and irradiation dosage was found in several studies (13, 30, 35).

The depolymerization of high-energy irradiated cellulose appears to be an oxidative degradation. Beta rays, gamma rays and thermal neutrons have all caused oxidation in various atmospheres, even in the absence of oxygen (5, 6, 7, 20, 37). Blouin and co-workers (4, 5, 6, 7) studied the degradation products of cotton after irradiation in several atmospheres. They found the presence of a large number of carbonyl groups (aldehydes or ketones) and some acid groups in the degraded fibres. Hydrogen, some carbon monoxide and carbon dioxide were evolved during the reaction. Carbonyl groups, acid groups and hydrogen were also observed by Emamura (16). The data of Blouin *et al.*(5) suggested but did not confirm that carbonyl groups were formed at C_1 and

C_4 with chain cleavage and also at C_2, C_3, C_5 and C_6 without chain cleavage and with production of hydrogen. Some of the carbonyl groups were further oxidized to acid groups. Blouin et al.(5) noted acid group production at chain ends, one group for every second chain cleavage. In one investigation, Teszler et al.(37) suggested that thermal neutrons caused only hydrolysis at early and late stages of irradiation, and oxidation during intermediate exposures.

During storage further decreases in viscosity have been noted. This "aftereffect" is believed to result from free radicals produced during the irradiation. These react with oxygen absorbed from the storage atmosphere, causing chain cleavage (21). Glegg (21) found that wood cellulose irradiated in nitrogen containing 0.26% moisture showed an aftereffect if stored in air or dry oxygen. However cotton (9, 22) and wood cellulose (22) containing at least 3.5% water vapour during exposure to gamma radiation in air showed little or no aftereffect when stored in air. The presence of sufficient water vapour during irradiation or storage therefore, curtailed any aftereffect in the storage atmosphere (21).

Authors have also investigated the possibility of changes in crystallinity. Infrared and X-ray spectroscopy analyses indicated that no decrease in crystallinity occurred with gamma-irradiated cotton fibres (4, 6). However an increased rate of dilute acid hydrolysis has suggested a reduction in crystallinity (4, 6, 35).

A number of the investigations on irradiated cellulose have been concerned with physical property changes. Tensile property changes, in particular decreased strength, have been attributed to random depolymerization of the cellulose molecule and decomposition of the anhydroglucose unit. Because high-energy radiations, especially gamma rays, can penetrate matter easily, crystalline and amorphous areas are affected equally. Recently, Krässig (26) has postulated that initially, chain ruptures in crystalline areas do not contribute to strength loss as these chains are held by hydrogen bonds. This is supported by the observation of a considerable decrease in viscosity before any appreciable strength loss is noted (2, 8, 38).

Generally, the decrease in strength observed was negligible at irradiation dosages up to 1.0×10^6 rads but evident and rapid at higher dosages (2, 4, 6, 19, 25). However gamma irradiation of cotton yarns and fibres has caused slight increases in strength between dosages of 0.88×10^5 and 0.93×10^5 rads (6, 31). Pan _et al_.(31) attributed this to a release of strain allowing a more even stress distribution within the fibre. Blouin and Arthur (8) found that cotton fibres and yarns exposed to a gamma ray dosage of 2.3×10^6 rads showed no significant reduction in strength. At 3.5×10^6 rads cotton yarns showed 20 to 30% loss in strength (18, 25, 38). A higher dosage,

4.4×10^6 rads, was required to cause a similar loss in cotton fibres (4, 6). Both cotton fibres and yarns lost 50% of their initial strength at 1.0×10^7 rads (2, 38) and 60% at 1.5×10^7 rads (38).

Viscose rayon filament yarns irradiated with gamma rays to 1.0×10^6 rads showed a slight gain in strength (2). Viscose staple yarns decreased 25% in strength at 3.5×10^6 rads (18) and 50% at 8.8×10^6 rads (19). Teszler et al. (38) observed a 25% strength loss in high tenacity rayon filament yarns irradiated with gamma rays to 1.5×10^7 rads.

Another tensile property, work required to break, also decreased after irradiation. Frank and Richards (18) observed a 19% loss for cotton thread and a 26% loss for viscose rayon staple yarn irradiated with gamma rays to 3.5×10^6 rads.

Teszler and co-workers (38) measured resistance to extension of irradiated cotton and rayon yarns. They observed that the modulus of cotton yarn calculated at the yield point of the stress-strain curve decreased 20% at 5.0×10^6 rads and slightly more, 22 to 23% at 1.5×10^7 rads. In the same study, high tenacity rayon yarn showed a 5% loss at 5.0×10^6 rads and no loss at 1.5×10^7 rads.

METHOD

SUMMARY

Random samples of each rayon conditioned at 65% relative humidity and 70°F., were placed in glass tubes and irradiated uncovered in air with gamma rays from Cobalt 60 in a Gammacell 220.[1] The dosages ranged from 0 to 3.0×10^7 rads at a dose rate of 1.21×10^6 rads/hour. The irradiated fibres were stored uncovered in air at 65% relative humidity and 70°F. until tested. Strength and initial modulus were evaluated from the tensile load-elongation curves of single fibres obtained from an Instron Tester. Instron integrator readings were recorded for calculation of the work to break. Linear density measurements were made on each fibre with an Insco vibroscope before breaking. These results were used to calculate strength in grams per tex. Twenty-five fibres were tested at each treatment level. Details of the test procedure follow.

FIBRES STUDIED

This study was confined to two staple rayon fibres, regular viscose and HM-64 modified viscose.[2] The staple fibre form was used since modified and polynosic rayons are

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[1]Manufactured by Atomic Energy of Canada Limited.

[2]One lb. representative samples of these rayons were supplied by Courtaulds (Canada) Limited.

used only in staple form. Initial properties of these fibres are listed in Table I.

TABLE I

INITIAL PHYSICAL PROPERTIES OF THE VISCOSE AND HIGH WET MODULUS RAYONS STUDIED

FIBRE	LINEAR DENSITY (Tex)	LENGTH (Inches)	INITIAL BREAKING TENACITY (Gm./Tex)	INITIAL MODULUS (Gm./Tex)
Regular Viscose	0.16*	1 5/8*	19.19	41.34
HM-64 Modified Viscose	0.16*	1 1/2*	34.74	64.09

* Nominal values provided by the manufacturer.

FIBRE SAMPLING

The viscose and high wet modulus rayon fibres were sampled according to the ASTM D540-64 zoning procedure (1) to obtain representative samples weighing approximately 3 grams. The two 3 gram samples were enclosed in bags made from a nylon filament fabric[3] and scoured as outlined in the B. S. Handbook No. 11, Appendix C, Method A (28) to remove impurities. The scoured samples were divided into 14 specimens, each weighing approximately 0.2 grams. The 0.2 gram specimens were placed in flat-bottomed glass tubes, 17 mm. in diameter and 65 mm. high.

[3]Fabric number 316, purchased from Testfabrics Incorporated, 55 Vandam Street, New York, New York, 10013.

IRRADIATION

The samples were conditioned at 65% relative humidity and 70°F. for at least 48 hours before irradiation. The circular test tube holder designed to fit the irradiation chamber of the Gammacell allowed all the tubes to be positioned at the same distance from the Cobalt 60. The holder had 24 circular slots arranged in two layers. The tubes were assigned randomly to the slots. Variations in irradiation intensity within the Gammacell chamber were considered negligible. The 14 specimens of each rayon allowed two replicates to be assigned at random to the following dosages:

level 0	0 rads
level 1	1.0×10^6 rads
level 2	2.0×10^6 rads
level 3	5.0×10^6 rads
level 4	1.0×10^7 rads
level 5	1.5×10^7 rads
level 6	3.0×10^7 rads

These treatment levels were chosen to give progressive degradation from 0 to 50% strength loss. Since Armstrong and Rutherford (2) found viscose rayon yarn strength increased slightly at 1.0×10^6 rads, this dosage was chosen as the lowest level. The samples were irradiated uncovered in air at a dose rate of 1.21×10^6 rads/hour.

EFFECT OF IRRADIATION ON TENSILE PROPERTIES

All samples were stored in the glass tubes uncovered at 65% relative humidity and 70^{o}F. for at least 48 hours prior to evaluation of tensile properties.

Breaking Strength and Work to Break

Breaking strengths were determined with the Instron Tester following the ASTM D540-64, Method B (1), using a test length range of 0.5 to 0.65 inches, an extension rate of 0.05 inches/minute, a chart speed of 2 inches/minute and a full scale deflection of 10 grams. Single fibres chosen at random were mounted slack between Insco vibroscope tabs, spaced 1/2 inch apart. Coloured opaque fingernail polish was used to attach the fibre ends to the tabs. The longer tabs, 60 mm. by 9.5 mm., were placed in the upper jaw of the Instron. The shorter tabs, 16 mm. by 9.5 mm., were clamped in the lower jaw.

Twenty-five results free of jaw breaks and slippage were obtained for each treatment. The fibres exposed to irradiation level 6 were not tested as they were too weak to support the weight of the short tabs during linear density measurements, or broke in handling.

Integrator readings were recorded for each break to allow calculation of work to break per unit length expressed in gm.cm./tex cm., according to the ASTM D2101-64T method (1). Work to break is equivalent to the area under the stress-strain curve as illustrated in Figure 1.

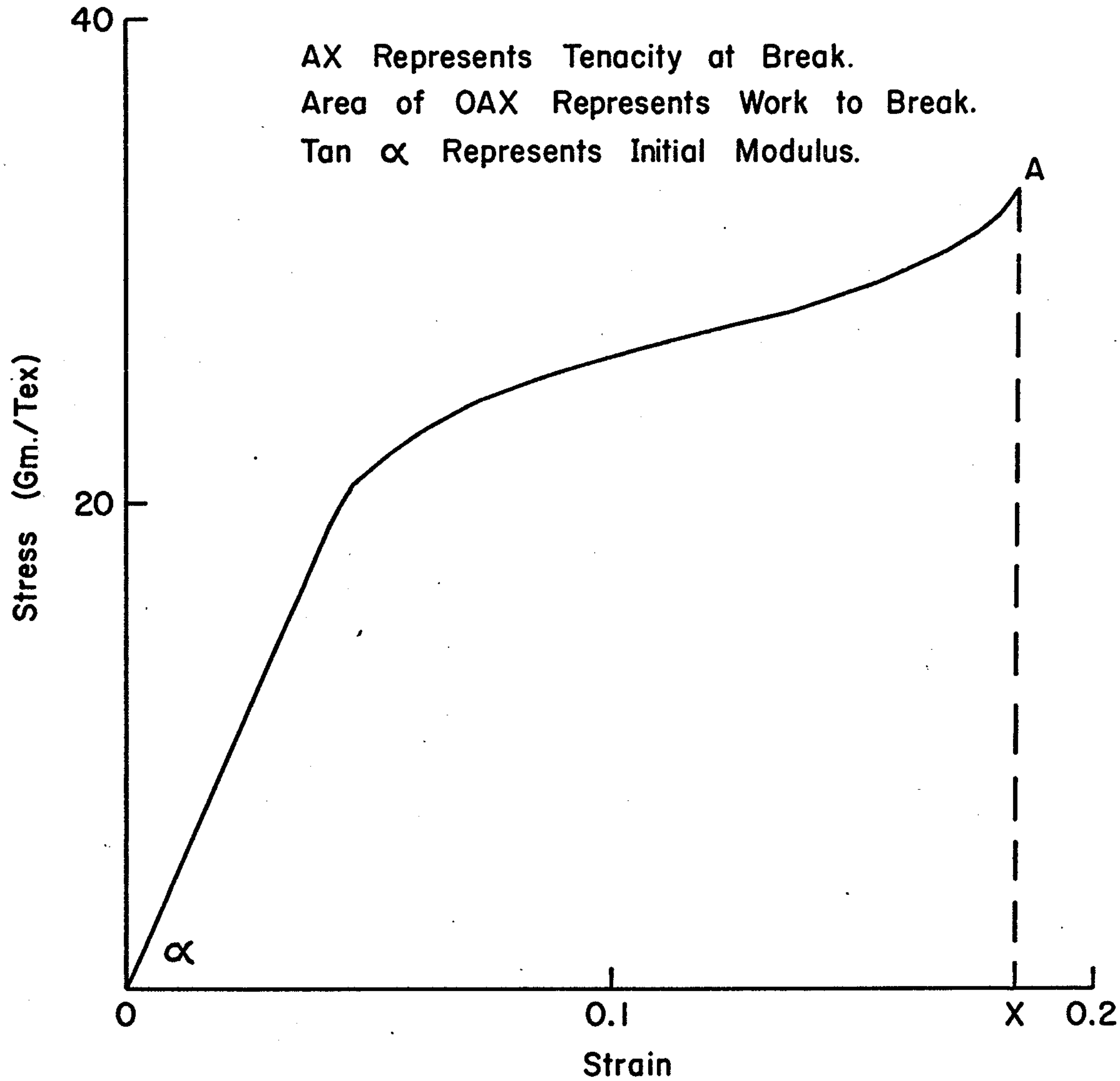

Figure I. Tensile Properties Expressed by a Typical Stress-Strain Curve.

Linear Density

Linear density measurements according to the ASTM D1577-66 Method A procedure (1) were made on all fibres before breaking, using an Insco vibroscope. The fibres were mounted as stated above. The shorter tabs weighing approximately 0.15 grams each provided sufficient tension to remove any crimp when the fibres were suspended from the vibroscope sample holder. Care was taken to put approximately the same amount of nail polish on the shorter tabs. After breaking, shorter tabs selected at random were weighed and showed no significant differences in weight.

These determinations were used to calculate the breaking tenacity in grams per tex.

Initial Modulus

The initial modulus was calculated following the ASTM D2101-64T method (1) from the initial straight line portion of the load-elongation curves as shown in Figure 1.

DERIVATION OF STRESS-STRAIN CURVES

Stress-strain curves were derived to allow graphical comparison of the effects of irradiation on the breaking characteristics of the rayons. The load-elongation curve of a fibre with breaking tenacity, work to break and initial modulus values closest to the mean values of these properties was selected as characteristic for each treatment. The loads at 0.0125, 0.025, 0.050 and 0.075 inches of fibre extension, the yield point and the breaking point were calculated in grams per tex. The representative stress-strain curves were plotted using these points.

STATISTICAL ANALYSIS

The twenty-five tests at each irradiation level were done consecutively. In all other respects, the experimental procedure was designed to be as completely random as possible. Coefficients of variation and 90% confidence intervals were computed for the means of the properties investigated.

Analyses of variance were made of both the breaking tenacity and work to break on the irradiation dosages. Estimated regression lines of these properties on irradiation dosages were derived.

RESULTS AND DISCUSSION OF RESULTS

The mean values of breaking tenacity, work to break, initial modulus and linear density, along with coefficients of variation and 90% confidence intervals are shown in Tables II and III. Stress-strain curves characteristic of each rayon at all treatment levels are presented in Figures 2 and 3.

The results of this study are reported in two forms: the percentage change in tenacity, work to break and initial modulus with irradiation for comparison with results from other studies, and the actual change in these properties for statistical analysis of the relationship between the property change and progressive irradiation.

BREAKING TENACITY

The breaking tenacities of both rayons decreased with increasing irradiation dosages.

Percentage loss in tenacity with irradiation dosage is illustrated in Figure 4. Both rayons showed a rapidly increasing strength loss with increasing irradiation up to 1.0×10^7 rads when the rate of loss levelled off. The results for the high wet modulus rayon were in good agreement with those reported by Armstrong and Rutherford (2), Frank and Richards (18) and Teszler _et al._(38) for gamma-irradiated cotton yarns.

TABLE II

PHYSICAL PROPERTY MEANS, COEFFICIENTS OF VARIATION AND 90% CONFIDENCE INTERVALS OF VISCOSE RAYON AT ALL IRRADIATION LEVELS

PROPERTY	IRRADIATION LEVEL	MEAN	COEFFICIENT OF VARIATION	90% CONFIDENCE INTERVAL
Breaking Tenacity (gm./tex)	0	19.19	14.88	18.25 - 20.13
	1	17.86	22.28	16.56 - 19.17
	2	17.59	15.52	17.04 - 18.14
	3	14.08	11.99	13.19 - 14.98
	4	9.35	26.37	8.54 - 10.16
	5	8.34	19.07	7.82 - 8.86
Work to Break (gm.cm./ tex cm.)	0	2.403	29.96	2.38 - 2.43
	1	2.286	36.75	2.01 - 2.56
	2	2.228	22.67	2.16 - 2.40
	3	1.562	25.61	1.43 - 1.69
	4	0.688	65.41	0.54 - 0.84
	5	0.606	56.11	0.49 - 0.72
Initial Modulus (gm./tex)	0	41.34	18.63	38.82 - 43.87
	1	54.06	17.37	50.98 - 57.14
	2	45.64	19.33	42.75 - 48.53
	3	47.44	14.92	45.12 - 49.76
	4	48.51	17.60	45.17 - 51.31
	5	48.52	23.58	44.77 - 52.27
Linear Density (tex)	0	0.183	10.00	0.177 - 0.189
	1	0.184	8.77	0.179 - 0.189
	2	0.181	8.23	0.176 - 0.186
	3	0.188	11.95	0.181 - 0.196
	4	0.185	10.05	0.179 - 0.191
	5	0.177	13.79	0.169 - 0.185

TABLE III

PHYSICAL PROPERTY MEANS, COEFFICIENTS OF VARIATION AND 90% CONFIDENCE INTERVALS OF HIGH WET MODULUS RAYON AT ALL IRRADIATION LEVELS

PROPERTY	IRRADIATION LEVEL	MEAN	COEFFICIENT OF VARIATION	90% CONFIDENCE INTERVAL
Breaking Tenacity (gm./tex)	0	34.74	19.69	23.52 - 45.96
	1	32.26	20.06	21.65 - 42.87
	2	30.18	15.28	22.62 - 37.74
	3	23.41	20.80	15.42 - 31.40
	4	14.54	31.28	7.08 - 22.00
	5	12.69	23.33	7.84 - 17.54
Work to Break (gm.cm./ tex cm.)	0	2.944	31.83	2.64 - 3.25
	1	2.597	24.64	2.39 - 2.81
	2	2.461	22.19	2.28 - 2.64
	3	1.510	33.77	1.34 - 1.68
	4	0.774	55.56	0.63 - 0.92
	5	0.550	52.73	0.45 - 0.65
Initial Modulus (gm./tex)	0	64.09	20.42	59.80 - 68.38
	1	54.38	21.88	50.48 - 58.28
	2	50.08	23.10	46.29 - 53.88
	3	68.21	20.26	63.68 - 72.74
	4	67.03	18.95	62.86 - 71.20
	5	56.72	15.60	53.82 - 59.62
Linear Density (tex)	0	0.185	14.70	0.176 - 0.194
	1	0.179	12.29	0.172 - 0.186
	2	0.175	19.60	0.164 - 0.186
	3	0.175	16.97	0.165 - 0.185
	4	0.199	18.39	0.187 - 0.211
	5	0.179	13.30	0.171 - 0.187

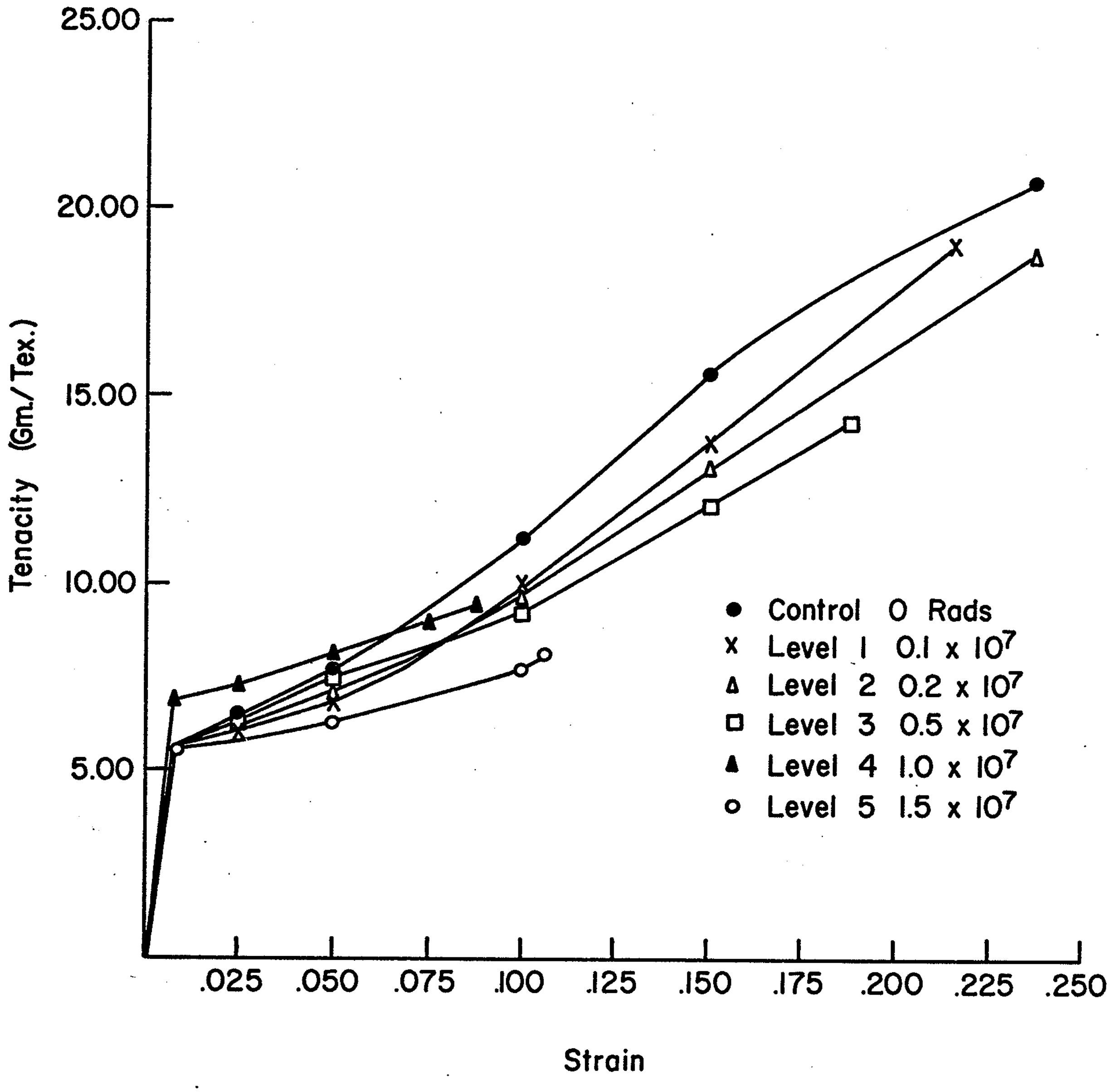

Figure 2. Stress - Strain Curves of Viscose Rayon Irradiated in Air with Gamma Rays.

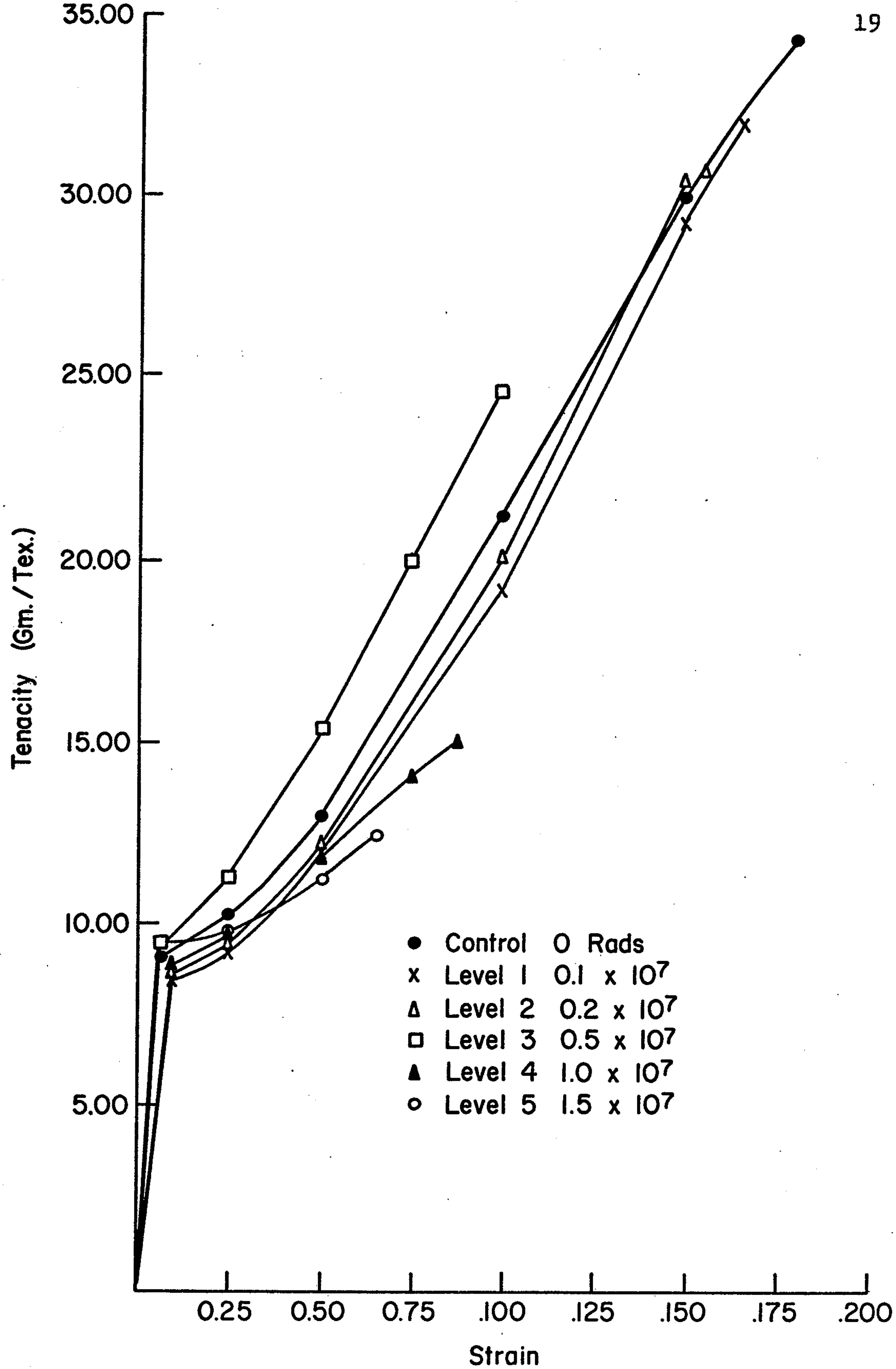

Figure 3. Stress-Strain Curves of High Wet Modulus Rayon Irradiated in Air with Gamma Rays.

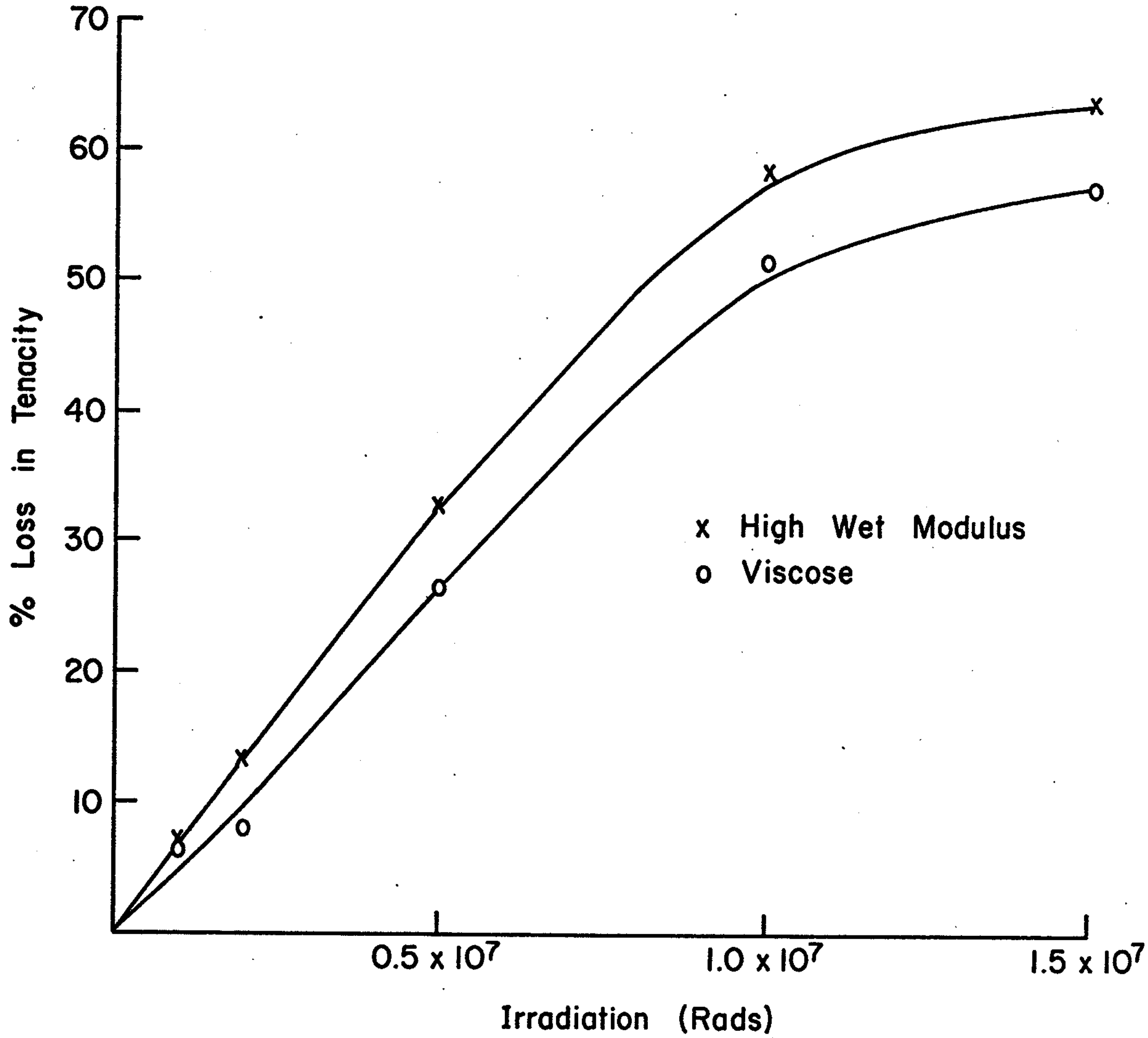

Figure 4. Percentage Loss in Tenacity of Viscose and High Wet Modulus Rayon with Irradiation.

The loss in tenacity for viscose rayon at 8.8×10^6 rads was consistent with the 50% loss found by Gilfillan and Linden (19). No increase was found at 1.0×10^6 rads as reported by Armstrong and Rutherford (2).

Mean breaking tenacities plotted against irradiation dosages for both rayons resulted in linear relationships as shown in Figure 5. Estimated regression lines for both rayons are presented in Table V. The slopes of the regression lines differed significantly from zero at the 5% level as shown by highly significant F test results in Table IV. The F tests for deviations gave slightly significant results indicating that the regression lines might not be linear. This is likely as it is characteristic of some kinds of degradation that the increase in degradation at any stage is proportional to the degradation that has already occurred and is represented by an exponential relationship. However, since the F values for regression were large in comparison with the F values for deviations, the slight significance shown in the F values for deviations was of no practical consequence and the relationships were considered linear.

The difference between regression slopes was found to be highly significant as shown in Table VI. This difference suggested that high wet modulus rayon had a higher rate of degradation than regular viscose on irradiation. A possible explanation for this is that only a few bonds between the large crystallites in high wet modulus

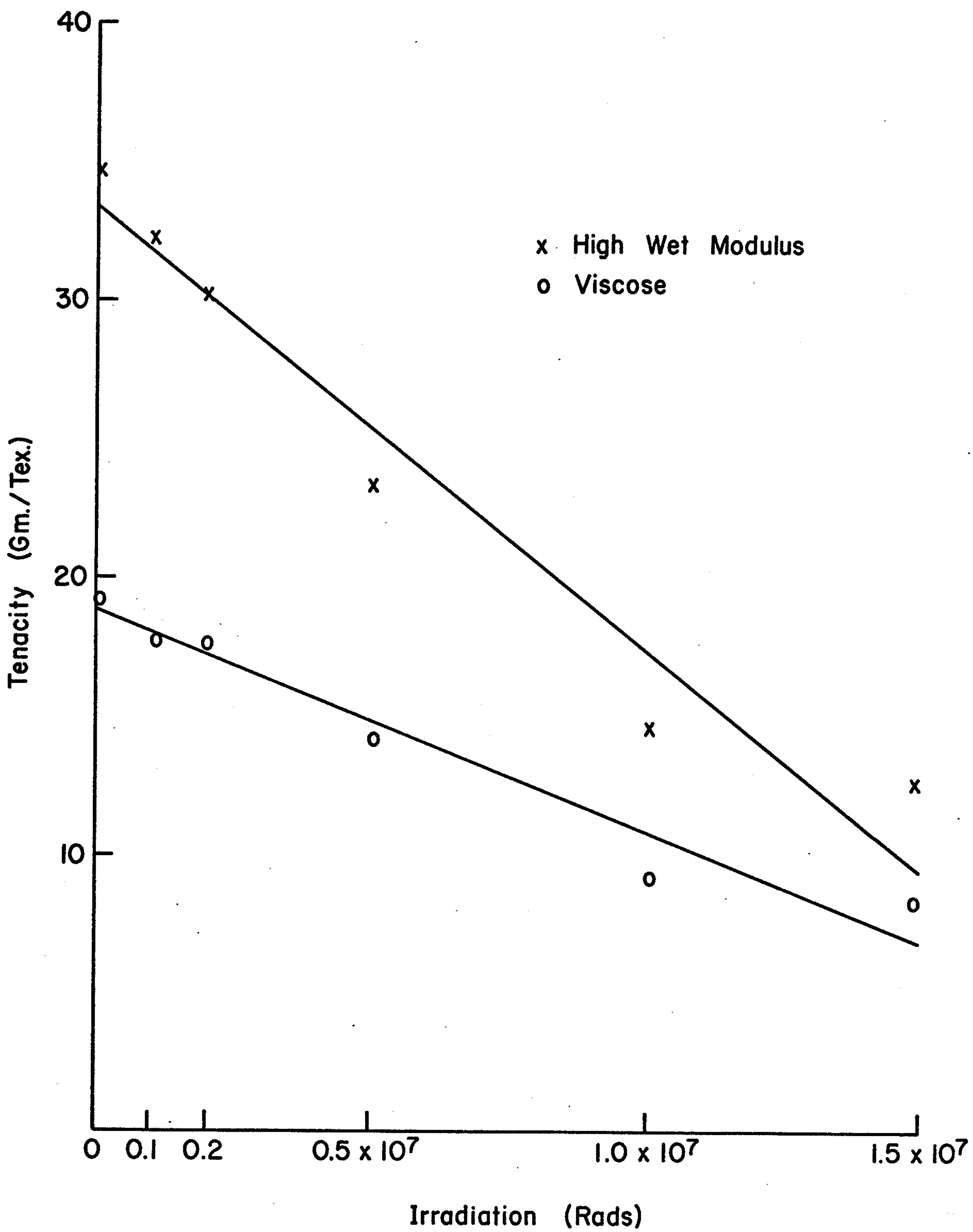

Figure 5. Decrease in Tenacity of Viscose and High Wet Modulus Rayon with Irradiation.

TABLE IV

ANALYSES OF VARIANCE OF TENACITY AND WORK TO BREAK ON IRRADIATION DOSAGES FOR BOTH RAYONS

TENACITY

FIBRE	SOURCE OF VARIATION	DEGREES OF FREEDOM	SUM OF SQUARES	MEAN SQUARE	F RATIO
VISCOSE	Treatments	5	2687.8960	537.5792	
	Regression	1	2559.0452	2559.0452	357.863*
	Residual	4	128.8508	32.2127	4.505*
	Error	144	1029.7332	7.1509	
	Total	149	3717.6292		
HIGH WET MODULUS	Treatments	5	10,929.0699	2185.8140	
	Regression	1	10,319.9516	10,319.9516	379.552*
	Residual	4	609.1183	152.2796	5.601*
	Error	144	3915.3325	27.1898	
	Total	149	14,844.4024		

WORK TO BREAK

FIBRE	SOURCE OF VARIATION	DEGREES OF FREEDOM	SUM OF SQUARES	MEAN SQUARE	F RATIO
VISCOSE	Treatments	5	84.4413	16.8883	
	Regression	1	78.5798	78.5798	725.739*
	Residual	4	5.8615	1.4654	4.210*
	Error	144	50.1321	0.3481	
	Total	149	134.5734		
HIGH WET MODULUS	Treatments	5	126.9787	25.3957	
	Regression	1	117.3249	117.3249	333.404*
	Residual	4	9.6538	2.4135	6.858*
	Error	144	50.6745	0.3519	
	Total	149	177.6532		

*Significant at the 5% level.

TABLE V

ESTIMATED REGRESSION EQUATIONS OF TENACITY AND WORK TO BREAK ON IRRADIATION DOSAGES FOR BOTH RAYONS

PROPERTY	FIBRE	EQUATION
TENACITY	VISCOSE	$y = 18.62 - 0.77 \times 10^{-6}x$
	HIGH WET MODULUS	$y = 33.44 - 1.60 \times 10^{-6}x$
		y represents tenacity x represents irradiation dosage
WORK TO BREAK	VISCOSE	$y = 2.36 - 0.14 \times 10^{-6}x$
	HIGH WET MODULUS	$y = 2.71 - 0.16 \times 10^{-6}x$
		y represents work to break x represents irradiation dosage

TABLE VI

TESTS OF SIGNIFICANT DIFFERENCES BETWEEN REGRESSION SLOPES

PROPERTY	DEGREES OF FREEDOM	CALCULATED[1] t	CRITICAL t
Tenacity	144	-9.351*	±1.97
Work to Break	144	-2.283*	±1.97

*Significant at the 5% level.

[1]Calculations were determined using the <u>Modified "Student's" t test</u>:

$$t = \frac{b_1 - b_2}{\sqrt{\dfrac{MS_1 + MS_2}{\sum x^2 - \frac{(\sum x)^2}{n}}}}$$

where b_1 and b_2 represent the slopes, MS_1 and MS_2 represent the mean square error terms,

and $\sum x^2 - \frac{(\sum x)^2}{n}$ represents the sum of squares of irradiation dosage.

rayon or cotton need to be broken for a great decrease in strength. With viscose, because of smaller crystallites and larger amounts of mechanically weak amorphous area, more bonds must be broken to bring about a comparable strength loss. A similar effect has been noted by Conrad et al.(15) when studying the tenacity of heat-degraded cotton and rayon.

WORK TO BREAK

Work to break of both rayons decreased rapidly with increasing irradiation. Percentage loss in work to break with irradiation is illustrated in Figure 6.

Mean work to break values plotted against irradiation dosages for both rayons gave linear relationships as shown in Figure 7. Estimated regression lines for both rayons are presented in Table V. The slopes of the regression lines differed significantly from zero at the 5% level as indicated by the highly significant F test results recorded in Table IV. The F values for regression were so large in comparison with the F values for deviations that the slight significance shown in the latter F values could be considered negligible.

The difference between regression slopes was shown to be significant as reported in Table VI. This difference led again to the supposition that the high wet modulus rayon had a higher rate of degradation.

The regression lines of work to break on irradiation

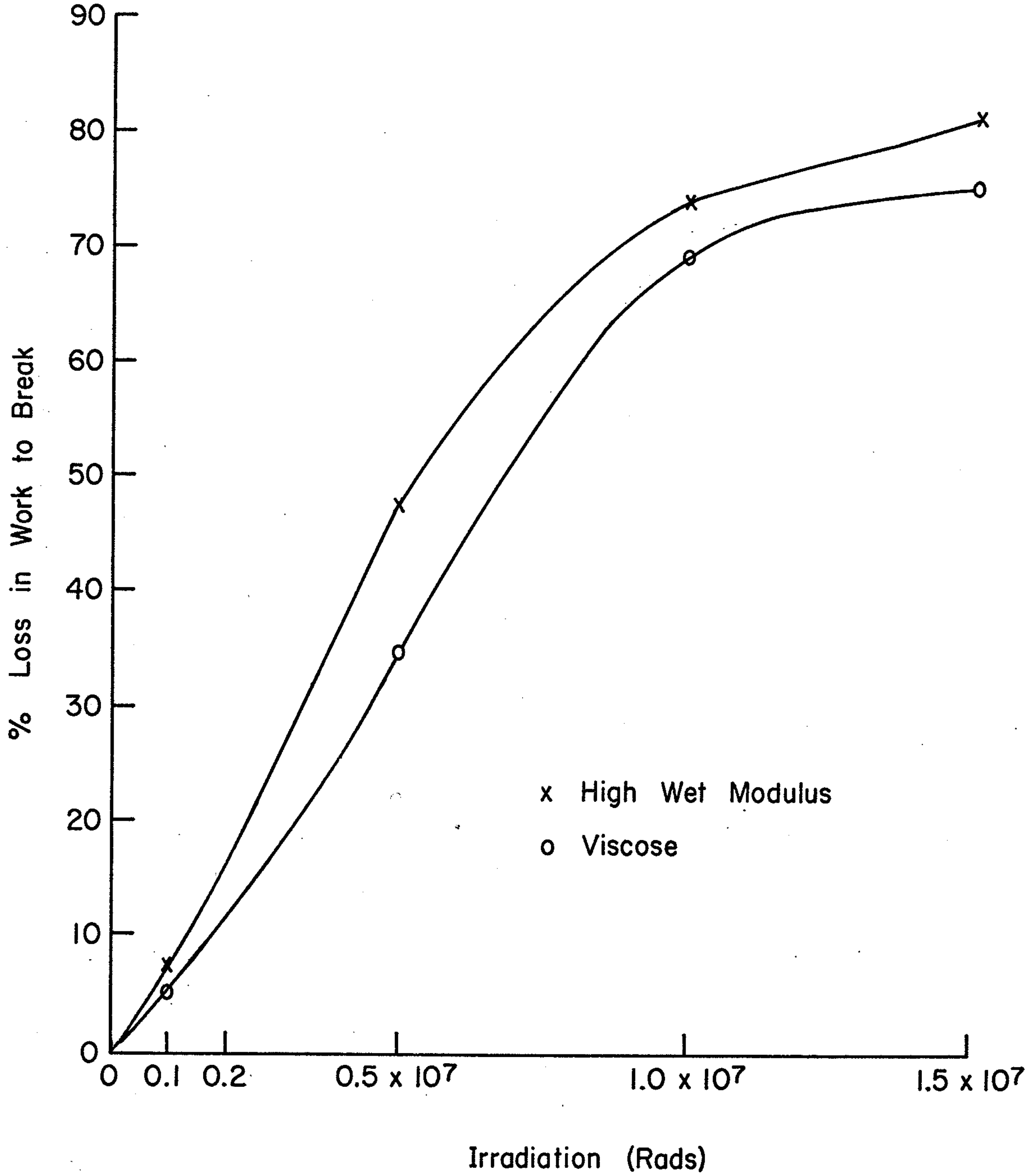

Figure 6. Percentage Loss in Work to Break of Viscose and High Wet Modulus Rayon with Irradiation.

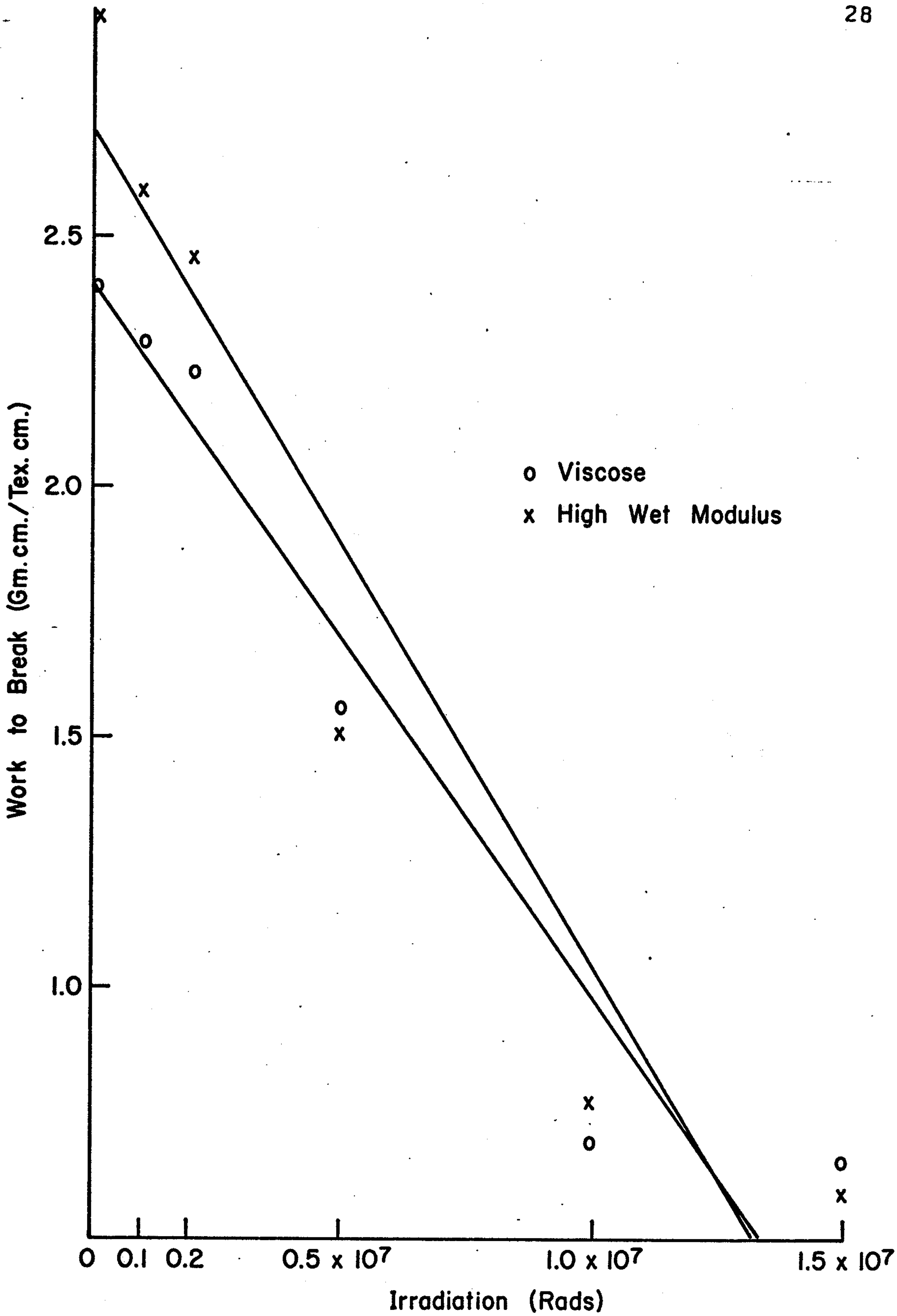

Figure 7. Decrease in Work to Break of Viscose and High Wet Modulus Rayon with Irradiation.

dosage had greater slopes than the regression lines of breaking tenacity plotted against irradiation. This suggests that work to break is affected more by irradiation than tenacity. This is reasonable as work to break is determined both by the breaking elongation of the fibre as well as the force required to break, whereas tenacity only indicates the force required.

AN EXPONENTIAL FIT TO THE DATA

The significant F test results for deviations suggested the possibility of an exponential relationship between tensile properties and progressive irradiation. Therefore, subsequent analyses of variance were carried out on the logarithms of breaking tenacity and of work to break on the irradiation dosages. The data fit the exponential relationship $y = y_0 10^{-bx}$ more closely than the former linear relationship. This better fit is indicated by the smaller F test results for deviations presented in Table VII. For tenacity, the F tests for deviations were slightly significant, but slightly smaller than those obtained in the former analysis. For work to break, the F tests for deviations were non-significant.

Estimated regression lines for both rayons are shown in Table VIII. The difference between the regression slopes of tenacity on irradiation was found to be significant as reported in Table IX. This agreed with the former analysis

TABLE VII

ANALYSES OF VARIANCE OF LOGARITHMS OF TENACITY AND WORK TO BREAK ON IRRADIATION DOSAGES FOR BOTH RAYONS

TENACITY

FIBRE	SOURCE OF VARIATION	DEGREES OF FREEDOM	SUM OF SQUARES	MEAN SQUARE	F RATIO
VISCOSE	Treatments	5	3.0808		
	Regression	1	2.9547	2.9547	350.9145*
	Residual	4	0.1261	0.0315	3.7411*
	Error	144	1.2125	0.0084	
	Total	149	4.2933		
HIGH WET MODULUS	Treatments	5	4.5643		
	Regression	1	4.3863	4.3863	312.6372*
	Residual	4	0.1780	0.0445	3.1718*
	Error	144	2.0200	0.0140	
	Total	149	6.5843		

WORK TO BREAK

FIBRE	SOURCE OF VARIATION	DEGREES OF FREEDOM	SUM OF SQUARES	MEAN SQUARE	F RATIO
VISCOSE	Treatments	5	12.3651		
	Regression	1	11.5892	11.5892	128.7689*
	Residual	4	0.7759	0.1940	2.1556
	Error	144	12.9595	0.0900	
	Total	149	25.3246		
HIGH WET MODULUS	Treatments	5	15.2268		
	Regression	1	14.7477	14.7477	174.6323*
	Residual	4	0.4791	0.1198	1.4186
	Error	144	12.1603	0.0845	
	Total	149	27.3871		

*Significant at the 5% level.

TABLE VIII

ESTIMATED REGRESSION EQUATIONS OF TENACITY AND WORK TO BREAK ON IRRADIATION DOSAGES FOR BOTH RAYONS

PROPERTY	FIBRE	EQUATION
TENACITY	VISCOSE	$y = (18.68)\ (10^{-2.61 \times 10^{-8} x})$
	HIGH WET MODULUS	$y = (33.34)\ (10^{-3.18 \times 10^{-8} x})$
		y represents tenacity x represents irradiation dosage
WORK TO BREAK	VISCOSE	$y = (1.92)\ (10^{-5.17 \times 10^{-8} x})$
	HIGH WET MODULUS	$y = (2.78)\ (10^{-5.83 \times 10^{-8} x})$
		y represents work to break x represents irradiation dosage

TABLE IX

TESTS OF SIGNIFICANT DIFFERENCES BETWEEN REGRESSION SLOPES

PROPERTY	DEGREES OF FREEDOM	CALCULATED[1] t	CRITICAL t
Tenacity	288	2.498*	±1.97
Work to Break	288	1.041	±1.97

*Significant at the 5% level.

[1]Calculations were determined using the "Student's" t test:

$$t = \frac{b_1 - b_2}{\sqrt{\dfrac{2\,Sp^2}{\sum x^2 - \dfrac{(\sum x)^2}{n}}}}$$

where b_1 and b_2 represent the slopes, Sp^2 represents the pooled estimate of the variance,

and $\sum x^2 - \frac{(\sum x)^2}{n}$ represents the sum of squares of irradiation dosage.

in suggesting that the high wet modulus rayon was more rapidly degraded than the regular viscose. However no significant difference between the regression slopes of work to break on irradiation was detected as shown in Table IX.

The greater slopes of the work to break regression on irradiation than of the tenacity regression on irradiation indicated that work to break was affected more by irradiation than tenacity. This supports the findings of the former analysis.

INITIAL MODULUS

Figure 8 shows the percentage change in the mean initial moduli of both rayons. There appears to be an increase in the initial modulus of viscose rayon with increasing irradiation. No clear trend was apparent with the initial modulus of high wet modulus rayon.

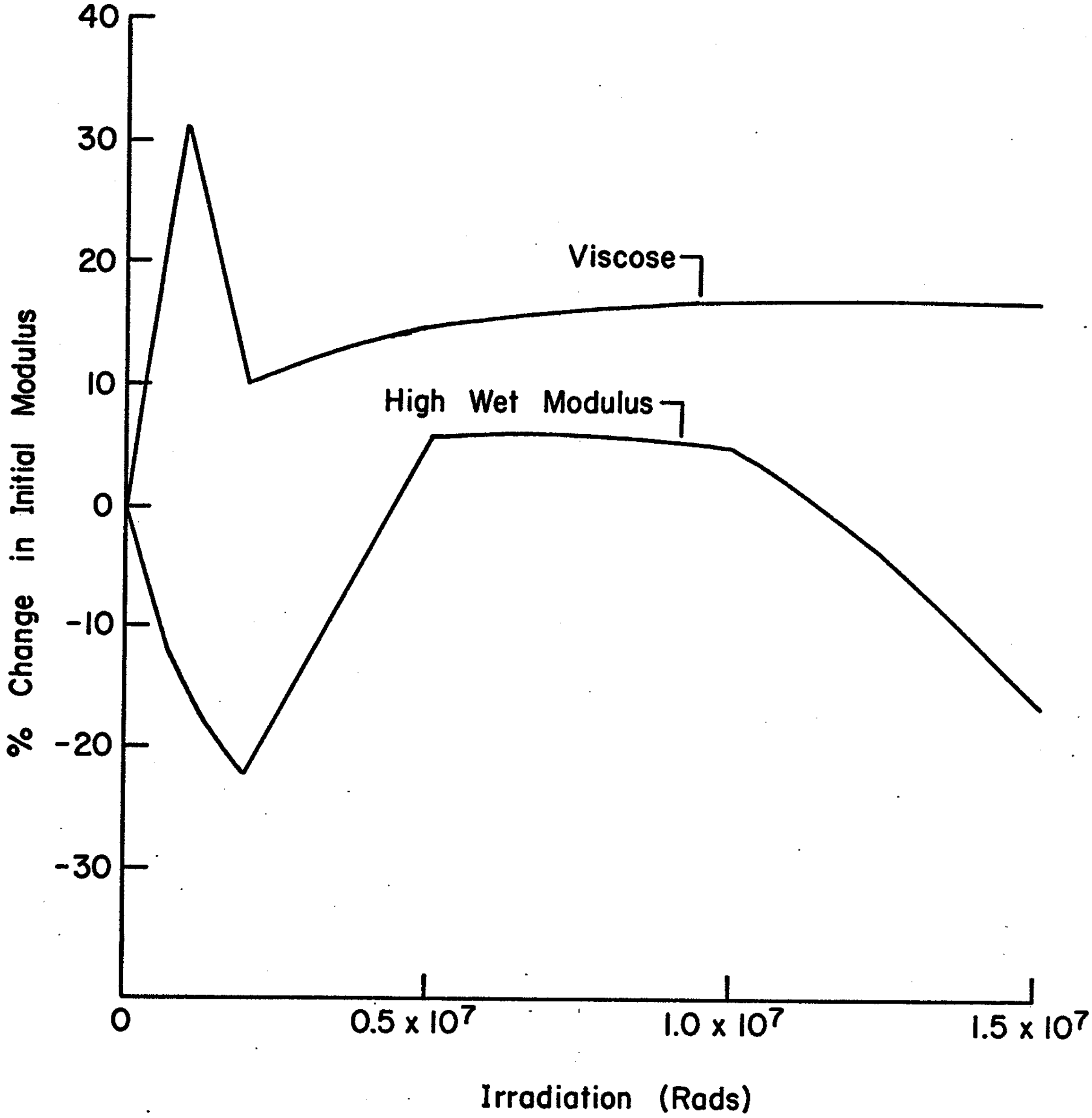

Figure 8. Percentage Change in Initial Modulus of Viscose and High Wet Modulus Rayon with Irradiation.

SUMMARY AND CONCLUSIONS

Viscose rayon and high wet modulus rayon staple fibres were irradiated uncovered in air with gamma rays from Cobalt 60 in a Gammacell 220. The seven dosages ranged from 0 rads to 3.0×10^7 rads at a dose rate of 1.21×10^6 rads/hour. Breaking strength and work to break measurements were made on 25 single fibres of each treatment. Initial modulus was measured on the load-elongation curves. All measurements were obtained using ASTM methods.

The fibres irradiated to 3.0×10^7 rads were too weak to handle.

Breaking tenacities decreased with increasing irradiation. The slopes of the regression lines of tenacities, and of the logarithms of tenacity, on irradiation dosages were significantly different from zero for both rayons. The high wet modulus rayon appeared to be more rapidly degraded as indicated by the slopes of the regression lines. Possibly, fewer bonds between the larger crystallites of high wet modulus rayon need be broken than between the smaller crystallites of viscose rayon for the same relative decrease in strength to occur.

Work to break decreased with increasing irradiation. Again the slopes of the regression lines of work to break and of the logarithms of work to break on irradiation dosages differed significantly from zero for both rayons. Work to break appeared to be more affected by irradiation than tenacity. This tendency was attributed to the fact that work required to break is dependent on the elongation in addition to the force necessary to break the fibres.

The initial modulus of the viscose rayon increased with increasing irradiation. There was no definite relation between the initial modulus of the high wet modulus rayon and irradiation dosage.

The results of this study show that high wet modulus rayons are comparable to cotton in their resistance to gamma rays. This indicates an additional similarity between the two types of cellulose.

For future study of the effects of gamma irradiation on high wet modulus rayons, the following considerations are recommended:

1) the use of intermediate irradiation dosages within the range of 0 to 1.5×10^7 rads. This might aid in determining the effect of irradiation on initial modulus;
2) the wet tensile properties of irradiated high wet modulus fibres;
3) the comparison of the breaking characteristics of irradiated high wet modulus fibres with those of irradiated cotton; and
4) the investigation of caustic alkali resistance and shrinkage resistance of irradiated high wet

modulus fibres.

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