Study of Creep, Stress Relaxation, and Inverse Relaxation in Mulberry (*Bombyx mori*) and Tasar (*Antheraea mylitta*) Silk

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ABSTRACT: The phenomena of creep, stress relaxation, and inverse relaxation/stress recovery were observed for mulberry and tasar silk. Instantaneous extension and secondary creep are both higher for tasar than for mulberry. The magnitude of inverse relaxation increases with the increase in peak tension and reduction in retraction for both varieties of silk. The extent of inverse relaxation was found to reduce because of cycling stressing. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 99: 3077–3084, 2006

Key words: *Bombyx mori*; *Antheraea mylitta*; thigh reeled tasar silk; creep; recovery; peak tension; inverse relaxation; cyclic stressing

INTRODUCTION

Textile materials are subjected to various types of forces during use. Tensile, compressive, flexural, torsional, and even their complex combinations are some typical examples of forces. All these forces lead to deformation which is time dependent in nature: when forces are withdrawn the time-dependent recovery phenomenon set in. These time-dependent deformation and recovery phenomena, known as creep and creep recovery, are therefore of great practical importance. During mechanical processing, yarns are subjected to different levels of strains for varying durations of time, while operating on loom and knitting machines. The interval between two successive changes in stress level will result in stress relaxation. During this relaxation process, the tension may either decrease (relaxation) or increase (inverse relaxation/stress recovery) depending upon the stress history.[1–13] Such a phenomenon has practical significance too: an increase in stress level may lead to occurrence of "weft bars" in the fabric during knitting and weaving operation as and when the process of fabric formation gets interrupted due to unavoidable stoppages for indefinite times.[2,14–16] Formation of such a bar would downgrade the quality of a fabric, especially for silk, since appearance is an extremely important characteristic for high quality silk fabric.

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Studies on creep of mulberry silk filaments were made in 1835 by Weber.[17] Leaderman[18] carried out numerous creep and creep-recovery tests on single filament of silk, at relatively low stresses. However, the investigation of this time-dependent behavior has not been reported for tropical tasar silk. Similarly, the phenomenon of inverse relaxation has been studied by researchers for spun yarns,[4–10] made from cotton, jute, acrylic, viscose, and polyester fibers, but not much work has been carried out for silk[19] even though it will be more crucial for silk fabric, since a neat and clean appearance is extremely important for high quality items.

Therefore, it was felt pertinent to study the phenomenon of creep for multivoltine mulberry (*Bombyx mori*) and tropical tasar silk (*Antheraea mylitta*) filaments and relaxation behavior of mulberry and tasar silk yarn, respectively.

EXPERIMENTAL

Materials

Multivoltine mulberry (*Bombyx mori*) and tropical tasar (*Antheraea mylitta*) silk cocoons were obtained from Central Silk Technological Research Institute, Bangalore, India and Raw Material Bank, Chaibasa, Bihar, India, respectively. Both the varieties of cocoons were cooked and reeled to obtain raw silk filaments of mulberry and tasar.

Commercial multivoltine mulberry and thigh reeled tropical tasar yarns were collected from different regional centers of the Central Silk Board, Bangalore, India.

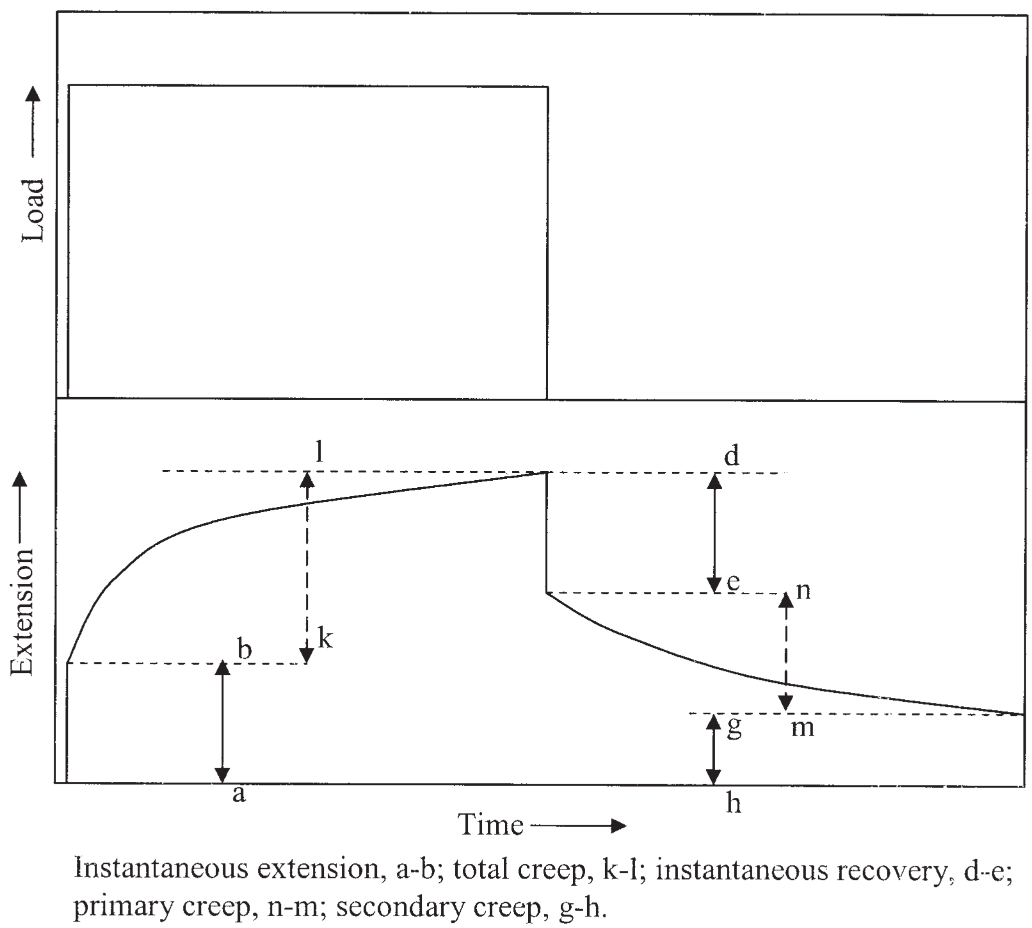

Figure 1 Schematic representation of creep and recovery. Instantaneous extension, a–b; total creep, k–l; instantaneous recovery, d–e; primary creep, n–m; secondary creep, g–h.

Methods

Multivoltine mulberry cocoons were taken in a wire mesh cage and cooked in the first pan at (65 ± 5)°C for 1 min, and then in the second pan at boiling for about one and half minute and finally at (65 ± 5)°C for 1 min. The cooked cocoons were hand brushed at boiling to a obtain true end of the filament. Because of the hard and compact nature of tropical tasar cocoons, those were cooked with 10% ethylenediamine at 80°C for 50 min. Deflossing of cocoons was done individually. The mulberry and tasar cocoons were then reeled on a wrap reel and continuous filaments were collected in the form of leas. Leas were degummed with 25% Marseilles soap (on the weight of the material) at the boil for 90 min, at a liquor ratio of 50:1. Degummed leas were washed, dried and conditioned at standard conditions of (27 ± 2)°C and (65 ± 2)% RH for 48 h. The filaments thus obtained were used for creep experiments. The tenacity and breaking extension were 2.34 cN/dtex and 13.5% for mulberry of 3.11 dtex fineness and 2.09 cN/dtex and 29.9% for tasar of 9.99 dtex fineness, respectively.

Samples selected for the relaxation study were commercially available mulberry (22.3 dtex) and thigh reeled tasar (149.3 dtex) yarns. The tenacity and breaking extension were 3.70 cN/dtex and 10.14% for mulberry and 1.45 cN/dtex and 21.43% for tasar silk, respectively. The values of yield stress were 52.9 cN and 112.6 cN, respectively, for mulberry and tasar yarns. All the samples were conditioned for 48 h prior to the determination of stress relaxation on an Instron Tensile Tester (model 1112), under (27 ± 2)°C and (65 ± 2)% RH.

Determination of creep

On application of a load to a fiber it will, after an instantaneous extension, continue to extend with time and, on removal of the load, the recovery will not be limited to the instantaneous recovery but will continue to take place. This behavior is illustrated in Figure 1 and is known as "creep" and "creep recovery". Figure 1 shows instantaneous extension, a–b; total creep, k–l; instantaneous recovery, d–e; primary creep, n–m; and secondary creep g–h.

The measurement of creep and creep-recovery of tasar and mulberry silk filament was carried out on a specially designed simple set up by suspending a 20

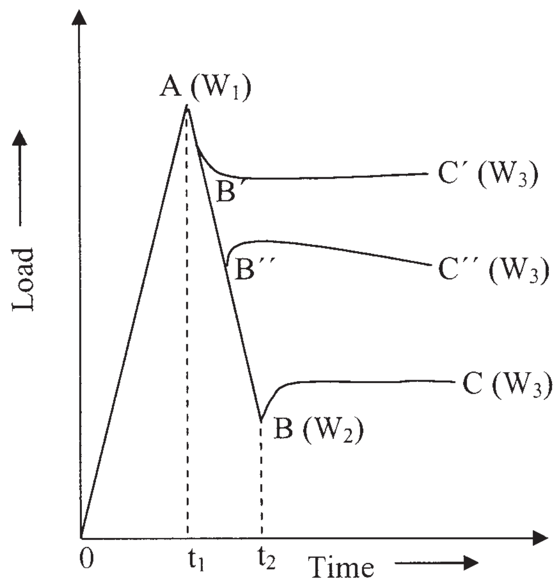

Figure 2 Schematic representation of inverse relaxation.

cm length of sample from a hook fixed to a wooden stand. The sample was given pretension by a paper clip. After taking the initial reading, predetermined loads (10 cN for tasar and 4 cN for mulberry, which are 60% of the breaking load) were suspended from the free end of the sample. The extension of the filament was measured by a Cathetometer at different intervals of time starting from 30 s onward till 60 min. After 60 min, the load was withdrawn from the sample, and the immediately contracted length was measured by adjusting the Cathetometer. The recovery that continued thereafter was measured at different intervals of time up to 60 min.

Determination of inverse relaxation

The phenomenon was studied following the procedure suggested by Nachane et al.[4] Figure 2 is a schematic representation of the procedure. A section of the yarn, held between the jaws of an Instron tensile tester (model 1112) was strained at a uniform rate "e" for a time t_1. The line OA represents the way load develops to a level W_1 as a result of extension $e\ t_1$. After attaining a load W_1, the jaw was reversed back for a time t_2, which caused the load to reduce following the line AB to a level W_2. The specimen was constrained at this level of extension by stopping the jaw movement. Now onwards, the way the load or the tension on the specimen changed was observed for a reasonable period of time until the change in load became negligible. Depending upon the situation, it was noticed that the load either increased following the line BC, decreased following the line B′C′; or showed a combined phenomena of initial rise followed by a fall following the line B″C″ to a level W_3. The ratio $[(W_3 - W_2)/W_1] \times 100$ has been taken as a measure of inverse relaxation index. A positive sign of the ratio would indicate a rise in tension, whereas a negative sign indicates a relaxation. The gauge length, chosen for both the yarns, was identical and was fixed at the level of 20 cm. The rate of straining was 10% of gauge length, that is, 2 cm/min. The selected value of peak tension (W_1) was 80, 60, and 40 cN for mulberry and 160, 120, and 80 cN for tasar yarn. For each level of peak tension (W_1), the levels of retraction values were varied, as shown in Table I.

Since yarns are subjected to repeated cyclic stress during weaving, the influence of repeated cyclic loading on the phenomenon of inverse relaxation was also studied. A schematic representation of the test procedure is shown in Figure 3.

After repeated cycling between two load levels T_1 and T_2, the tension level was retracted to a level T_3 during downward stroke of the cycle. Now onwards, the change in tension level was observed. The number of load cycles chosen was 40. The level of peak tension was 80 and 160 cN for mulberry and tasar yarns, respectively, and the level of retraction was 70, 54, and 30 cN for mulberry and 140, 114, and 60 cN for tasar yarn.

RESULTS AND DISCUSSION

Creep and recovery of silk filament

The results of creep experiments were plotted and are demonstrated in Figure 4 and Table II. It is observed that the total extension is much larger for tasar than

TABLE I
Retraction of Various Peak Tension Levels

Mulberry		Tasar	
Peak tension (cN)	Level of retraction (cN)	Peak tension (cN)	Level of retraction (cN)
80	70	160	140
	64		130
	54		119
	40		114
	30		96
	20		60
	10		40
			20
			10
60	50	120	100
	40		80
	30		60
	20		40
	10		20
40	30	80	60
	28		56
	20		40
	10		20
	9		

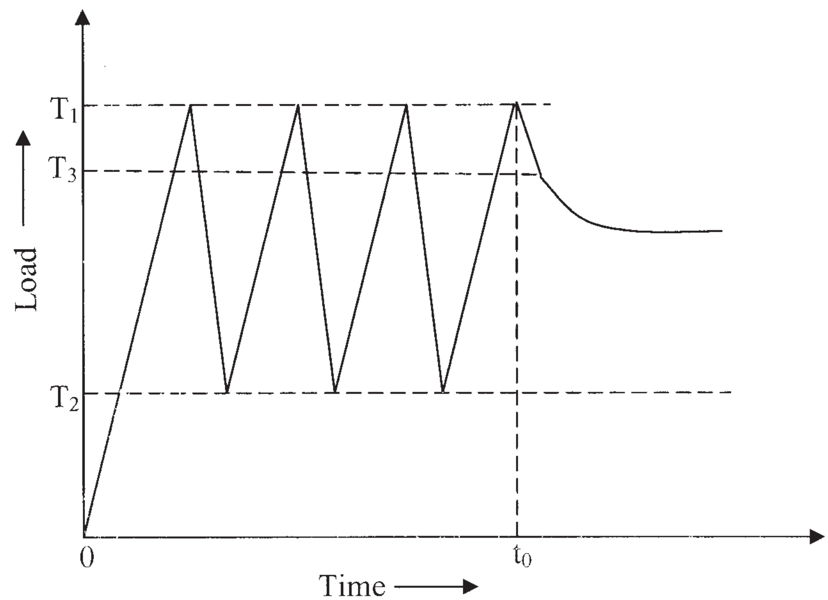

Figure 3 Schematic representation of inverse relaxation after cyclic loading.

for mulberry. In the total extension, the creep component is more or less similar for mulberry and tasar, that is, around 5%.

The instantaneous extension is, however, much larger in tasar. As far as the recovery behavior is concerned, a great difference is observed especially in the values of secondary creep, which is 7.5% for mulberry and 19% for tasar. The instantaneous recovery and primary creep values are slightly higher in the case of tasar.

Such a difference in the deformation and recovery behavior can be attributed to the structural differences of the two varieties, that is, mulberry and tasar. Tasar has a highly disordered structure manifested by the lower density, birefringence, orientation index, and

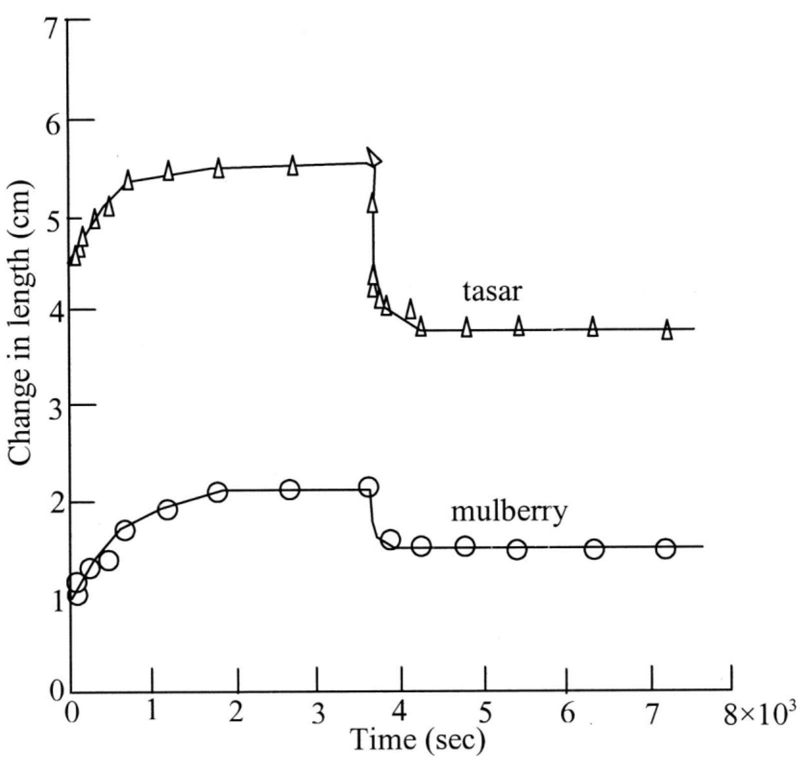

Figure 4 Creep and recovery of mulberry and tasar filament.

TABLE II
Deformation and Recovery of Mulberry and Tasar Filament

Variety	Instantaneous extension (%)	Total creep (%)	Total extension (%)	Instantaneous recovery (%)	Primary creep (%)	Secondary creep (%)
Mulberry	5.0	5.5	10.5	2.5	0.5	7.5
Tasar	22.5	5.0	27.5	6.0	2.5	19.0

sonic modulus.[20] In addition, tasar has a higher percentage of bulky side groups, as shown by Lucas et al.,[21] which will induce easy flow under the application of load. Hence, when such a structure is loaded, both the instantaneous extension and secondary creep are expected to be higher.

Influence of level of retraction on inverse relaxation behavior of silk yarn

The results depicted in Figures 5 and 6 demonstrate that, at any level of tension, there exists a possibility for inverse relaxation to occur depending upon level of retraction for both mulberry and tasar yarns. Inverse relaxation increases with the increase in peak tension level for both the yarns. It will be interesting to know what the level of retraction for different tension level has to be for the manifestation of inverse relaxation phenomena. To estimate this, vertical lines were drawn from the crossover points of inverse relaxation–retraction curves and zero inverse relaxation line to the x-axis indicating retraction. The points at which the vertical lines cut the x-axis were read and recorded, as shown in the Table III. It may be observed

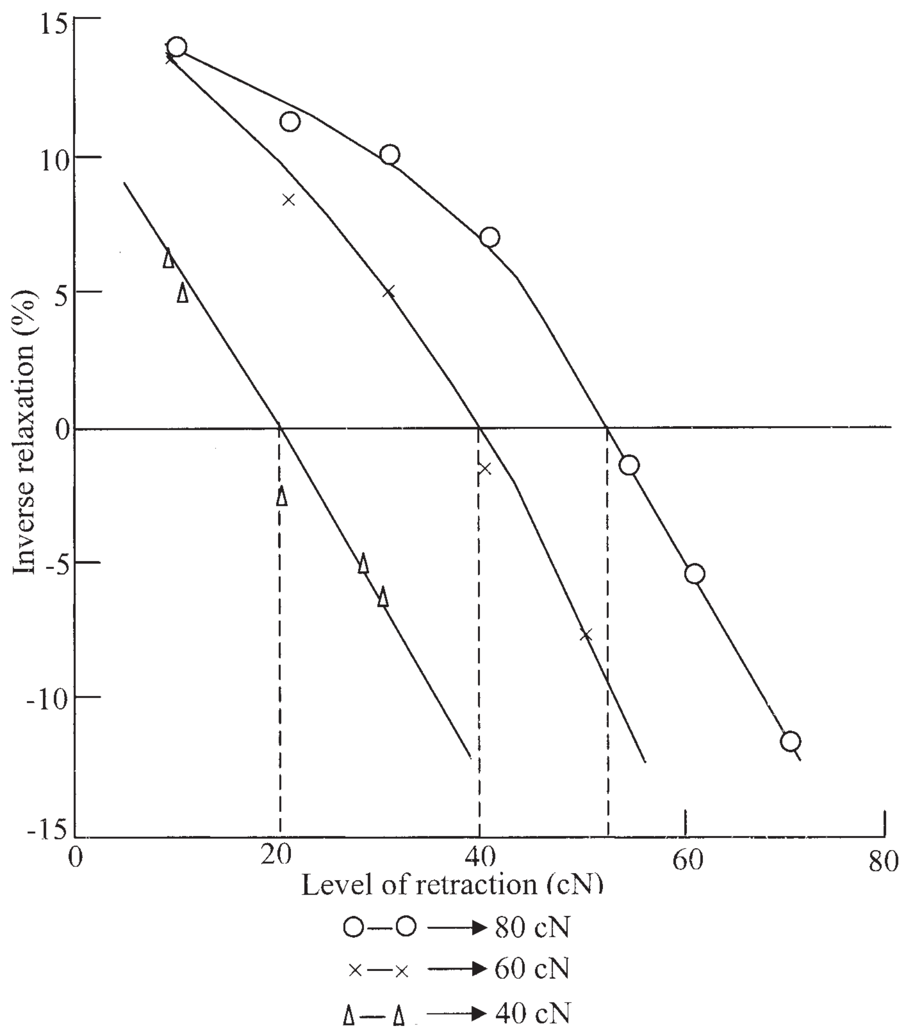

Figure 5 Effect of level of retraction on inverse relaxation for mulberry silk yarn. (○): 80 cN; (×): 60 cN; (△): 40 cN.

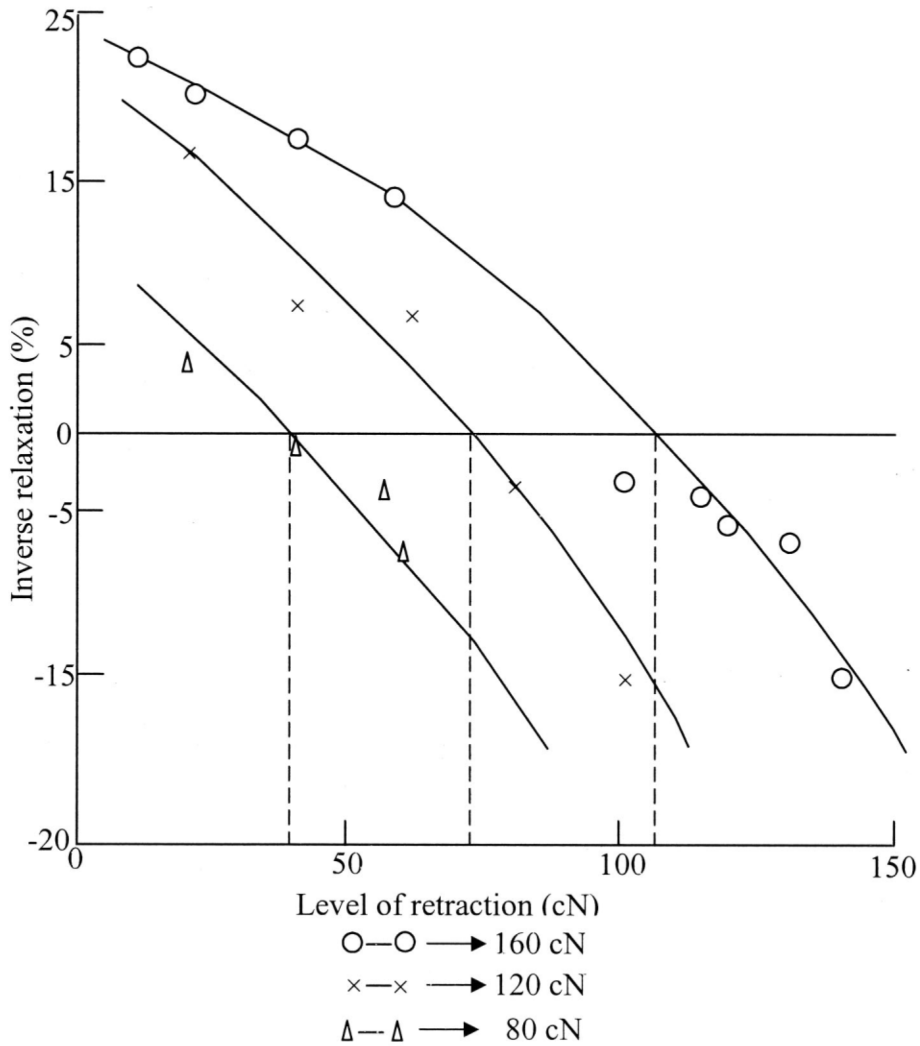

Figure 6 Effect of level of retraction on inverse relaxation for tasar yarn. (○): 160 cN; (×): 120 cN; (△): 80 cN.

from Table III that, when the peak tension level is kept beyond yield stress, a level of retraction equivalent to 66% of peak tension level or below is needed to observe the phenomena of inverse relaxation for both the yarns. However, when the tension level is below the yield stress, that is, 40 cN for mulberry and 80 cN for tasar, retraction even below 50% is likely to show the inverse relaxation.

TABLE III
Level of Retraction Corresponding to Zero Inverse Relaxation for Various Peak Tension

Type of yarn	Peak tension (cN)	Retraction corresponding to zero IR (cN)	Retraction corresponding to zero IR (%)
Mulberry	80	53	66.2
	60	40	66.0
	40	20	50.0
Tasar	160	106.5	66.0
	120	75.0	62.5
	80	40.0	50.0

IR, inverse relaxation.

The phenomenon of inverse relaxation can be explained, as stated by Nachane and Sundaram[9] Silk, being a viscoelastic material, when stretched to a certain level, the extension takes place through stretching of lateral intermolecular bonds and subsequent slippage of molecules.

As it is retracted, the process reverses, in the sense that the molecular slippage now takes place in the reverse direction and hence, even though the retraction is discontinued by stopping the reverse movement of jaws of Instron, the process of molecular slippage in the reverse direction continues, leading to a rise in tension.

Influence of cyclic loading on inverse relaxation behavior of silk yarn

The phenomenon of inverse relaxation was also found to take place, even after cyclic loading of the specimen

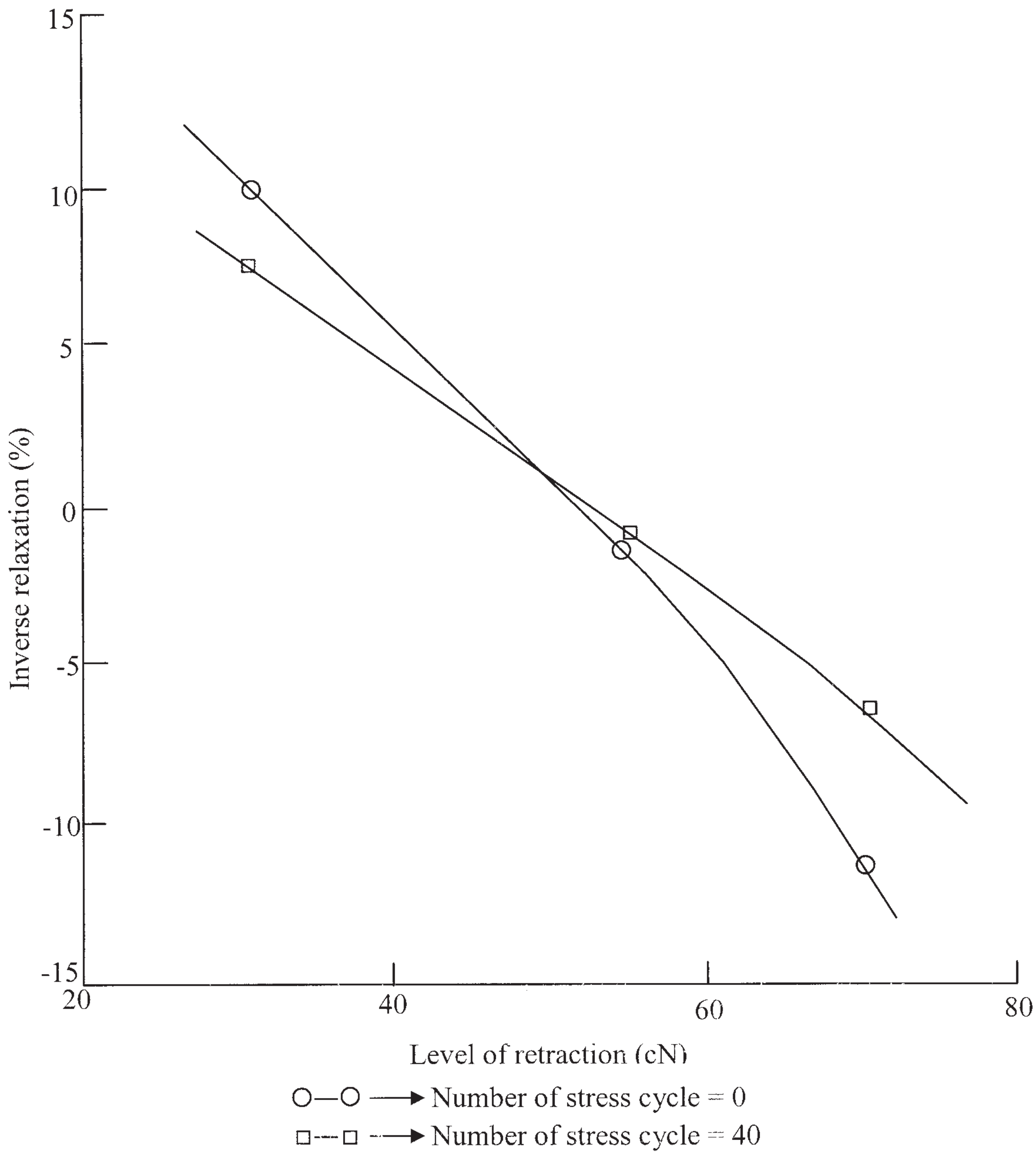

Figure 7 Effect of level of retraction on inverse relaxation for mulberry silk yarn after cyclic loading. (○): Number of stress cycle = 0; (□): number of stress cycle = 40.

between two load limits (80 and 30 cN for mulberry and 160 and 60 cN for tasar). However, the inverse relaxation index was less for both cyclically loaded mulberry and tasar yarns, as shown in Figures 7 and 8. Repeated cyclic stressing brings the material to a stable condition, and as a result it relaxes less during relaxation period, as stated by Vangheluwe and Kiekens.[5]

CONCLUSIONS

Under equivalent loading condition, that is, a fixed percentage of breaking load, the deformation and recovery behaviors are different in tasar and mulberry filament. Instantaneous extension and secondary creep are both higher for tasar than for mulberry. Secondary creep, being larger in tasar, the shape retention of any product of tasar is expected to be inferior than that of mulberry.

Inverse relaxation phenomena were observed for both mulberry and tasar yarns, only when the level of retraction was maintained below a certain value, becoming higher with the increase in peak tension and reduction in retraction. The extent of inverse relaxation was found to reduce as a result of cyclic stressing.

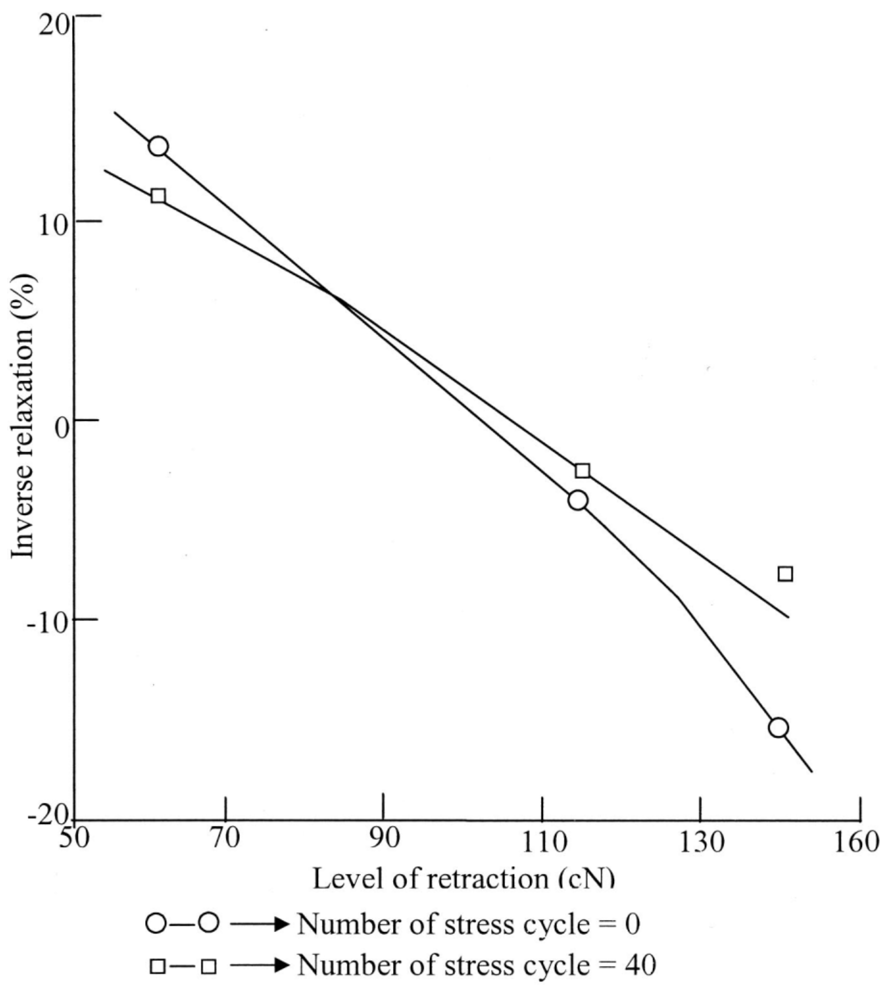

Figure 8 Effect of level of retraction on inverse relaxation for tasar silk yarn after cyclic loading. (○): Number of stress cycle = 0; (□): number of stress cycle = 40.

References

1. Greenwood, K.; Cowhig, W. T. J Text Inst 1956, 47, T241.
2. Stein, H.; Weyden, H. V. Textilindustrie 1968, 70, 554.
3. Wulfhorst, B.; Obolenski, B. Melliand Textilber 1990, 71, 24.
4. Nachane, R. P.; Hussain, G. F. S.; Patel, G. S.; Krishna Iyer, K. R. J Appl Polym Sci 1986, 31, 1101.
5. Vangheluwe, L.; Kiekens, P. Melliand Textilber 1992, 73, 117.
6. Manich, A. M.; De Castellar, M. D. Text Res J 1992, 62, 196.
7. Nachane, R. P.; Hussain, G. F. S.; Krishna Iyer, K. R. Text Res J 1982, 7, 483.
8. Nachane, R. P.; Hussain, G. F. S.; Patel, G. S.; Krishna Iyer, K. R. J Appl Polym Sci 1989, 38, 21.
9. Nachane, R. P.; Sundaram, V. J Text Inst 1995, 85, 10.
10. Nachane, R. P.; Sundaram, V. J Text Inst 1995, 86, 20.
11. Bhuvanesh, Y. C.; Gupta, V. B. Polymer 1995, 36, 3669.
12. Hussain, G. F. S.; Nachane, R. P. J Appl Polym Sci 2001, 81, 2563.
13. Nachane, R. P.; Hussain, G. F. S. Ind J Fibre Text Res 2003, 28, 50.
14. Vitkauskas, A.; Matukonis, A. Techn Text Ind USSR 1968, 4, 21.
15. Greenwood, K. J Text Inst 1956, 47, 241.
16. Inui, H. Text Machinery Japan 1969, 1, 85.
17. Weber, W. Pegg Ann Physik 1835, 4, 247.
18. Leaderman, H. The Textile Foundation; Washington, DC, 1943.
19. Kothari, V. K.; Rajkhowa, R.; Gupta, V. B. J Appl Polym Sci 2001, 82, 1147.
20. Das, S.; Chattopadhyay, R.; Gulrajani, M. L.; Sen, K. 3rd Indo-Czech Text Res Conf, Czech Republic 2004, 150.
21. Lucas, F.; Shaw, J. T.; Smith, S. G. J Text Inst 1955, 46, 440.