Study on the Viscoelastic Response of Silk

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SYNOPSIS

Simple stress–strain characteristics of silk do not give sufficient information on the influence of the rate of testing in silk. A methodology is described to quantify individual components of viscoelasticity which clearly brings out the influence of the strain rate on the viscous component of the system. © 1996 John Wiley & Sons, Inc.

INTRODUCTION

Various physical properties of silk were studied with renewed interest to obtain fibers with improved properties. Chemical modifications including grafting with polymers were carried out to improve handling and decrease photoyellowing.[1–3] These modifications influence the mechanical properties, which do have a major role in deciding the usefulness of such modifications. Being a viscoelastic fibrous biopolymer, any change in the degree of crystallinity affects the mechanical properties of silk.[4,5] Therefore, prior to examining the effect of chemical modifications, a better understanding of the viscoelastic behavior of silk is essential. While earlier studies were restricted to the tensile strength and hysteresis behavior of silk,[6] quantification of various viscoelastic components, which would give better insight into the tensile behavior of silk, is yet to be carried out. In the present study, an attempt was made to quantify the viscous, elastic, and plastic energy components with reference to the strain rate.

EXPERIMENTAL

Raw silk obtained from cocoons produced in India (*Bombyx mori*) were made into silk filature yarn with an 8–10 TPI twist (23.625 den). These undegummed fibers were conditioned at 20°C and 65% RH and then tested using an Instron 1112 at various strain rates. The gauge length was set to 10 cm. The strain rates used were 2, 10, 20, 50, 100, and 200% per min. By modifying the procedure suggested by Naresh et al.,[7] various components like elastic en-

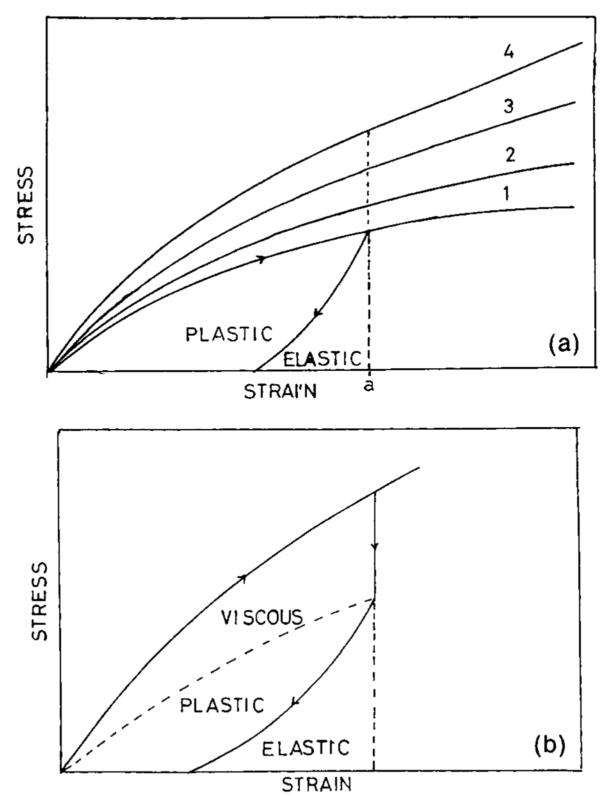

Figure 1 (a) Effect of strain rate on the conventional method of computing elastic and plastic energies. 1–4 represent stress–strain curves at different strain rates (1 < 2 < 3 < 4). (b) Modified method of determining the elastic, plastic, and viscous energies.

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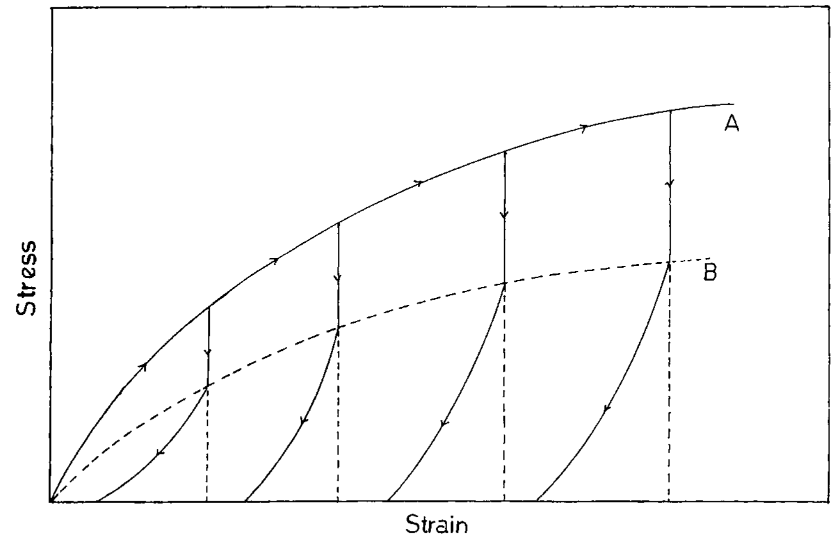

Figure 2 Deriving the time-independent stress–strain curve: (A) experimental stress–strain plot; (B) time-independent stress–strain curve (strain rate approximately 0% per min).

ergy, viscous energy, and plastic energy are determined. The method is briefly described below.

In an experimental stress–strain curve (for a viscoelastic material), the stress registered at a given strain level is time-dependent as it will increase with increasing strain rate. For a given strain level, if elastic energy is determined by the conventional method—i.e., conducting the hysteresis experiment and measuring the area outside the loop—it would vary with the rate of testing [Fig. 1(a)]. This would make the elastic component time-dependent, which is not true. Hence, extending a sample to a given strain level (irrespective of the strain rate) and allowing the stress developed to decay at that point until the equilibrium is reached and then completing the hysteresis cycle would not only give a better quantification of elastic and plastic energies, but, also, in the process, give a measure of the viscous component [Fig. 1(b)]. Repeating the above methodology at various strain

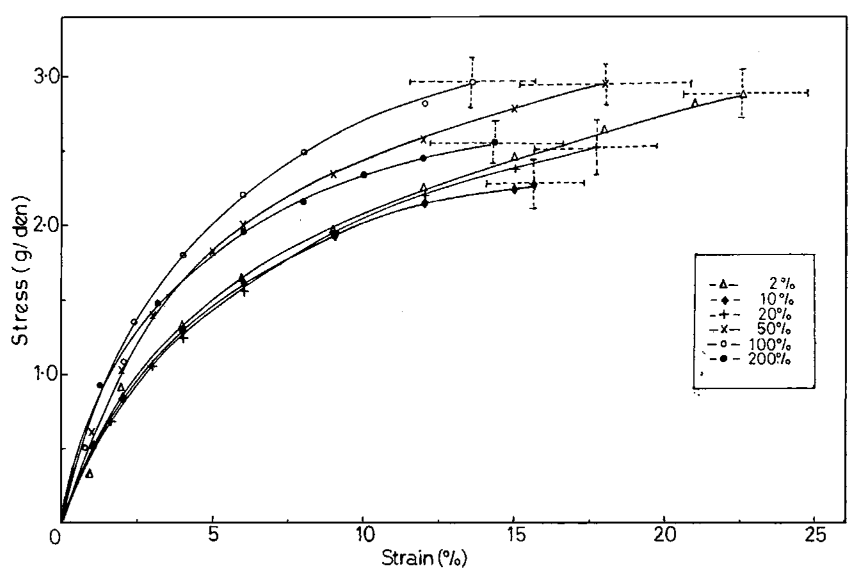

Figure 3 Stress–strain characteristics of silk at different strain rates.

Table I Plastic Set at Different Strain Rates (Percent)

	Strain Rate					
Strain	2%	10%	20%	50%	100%	200%
3%	1.4	1.4	1.1	0.9	0.8	1.3
6%	3.2	3.5	3.3	2.4	2.8	4.0
9%	5.8	6.3	5.7	5.7	4.8	5.8
12%	8.0	8.2	8.0	8.1	8.0	8.7
15%	10.4	10.7	11.0	10.8		
18%	12.8					

levels would help in reconstructing a time-independent stress–strain curve (0% strain rate) within experimental limits [Fig. 2].

Thus, in the present study, the stress–strain characteristics of silk were determined for different strain rates initially and then another set of experiments were done using the modified methodology given below. Specimens were extended to predetermined strain levels and relaxed until the stress decayed to a steady level and then the samples were taken back to the zero-stress level. Graphs similar to Figure 2 were plotted after normalizing the stress reached in individual relaxation cum hysteresis experiments (for different strain levels) to the stress observed in the stress–strain plots. The enclosed areas under different regions were measured and the energy calculated in MJ/m^3 after converting the g/den stress values to MPa using the density of silk.[5]

Table II Energy Components (MJ/m^3)

	Strain Rate					
Strain	2%	10%	20%	50%	100%	200%
Viscous energy						
3%	0.978	0.835	0.787	1.025	1.431	1.860
6%	2.432	2.194	2.194	3.099	3.791	4.578
9%	3.958	3.863	4.101	5.651	6.724	7.630
12%	5.818	5.842	6.295	8.679	9.728	10.777
15%	7.602	7.535	8.703	11.993		
18%	9.919					
Elastic energy						
3%	0.644	0.477	0.548	0.715	0.811	0.548
6%	1.359	1.192	1.311	2.814	1.812	1.121
9%	1.908	1.621	1.645	2.098	2.432	1.980
12%	2.623	2.265	2.027	2.432	3.410	2.051
15%	3.386	2.671	2.480	2.480		
18%	4.053					
Plastic energy						
3%	1.025	0.906	0.858	0.715	0.954	0.858
6%	3.910	3.553	3.267	3.910	4.220	3.934
9%	8.059	7.868	7.105	8.655	9.132	7.535
12%	13.043	12.518	12.017	13.949	14.640	12.637
15%	18.789	18.217	17.215	20.124		
18%	24.988					

RESULTS AND DISCUSSION

The stress–strain characteristics of silk yarn at different strain rates are presented in Figure 3. It is evident that the mechanical response of silk to the strain-rate variation is very minimal when compared to other fibrous biopolymers like collagen or elastoidin.[8,9] Earlier studies on the tensile properties of silk have also shown that there was only an increase of 10–15% for increasing the strain rate from 12 to 300% per min.[6] When dynamic mechanical characteristics were studied, the increase was 32% for increasing the rate of testing from 0.028 to 170 Hz.[10] The plastic set developed in the system due to cycling at various strain rates also does not clearly bring out the effect of strain rate (Table I).

However, using the modified methodology, if the various components that contribute to the viscoelastic response of silk is taken into consideration,

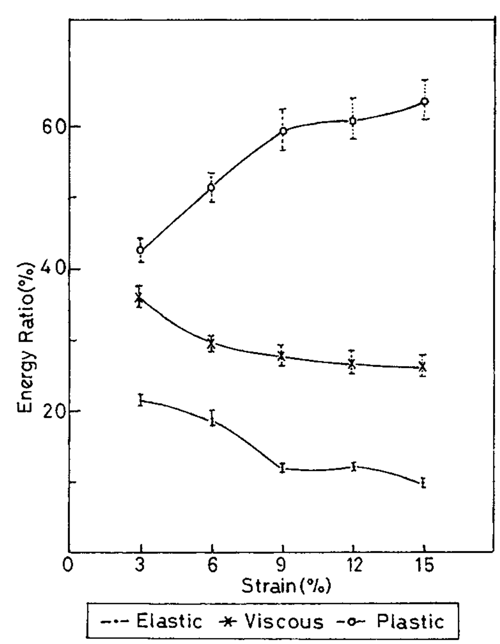

Figure 4 Percent energy contribution of viscous, elastic, and plastic components (strain rate is 10% per min).

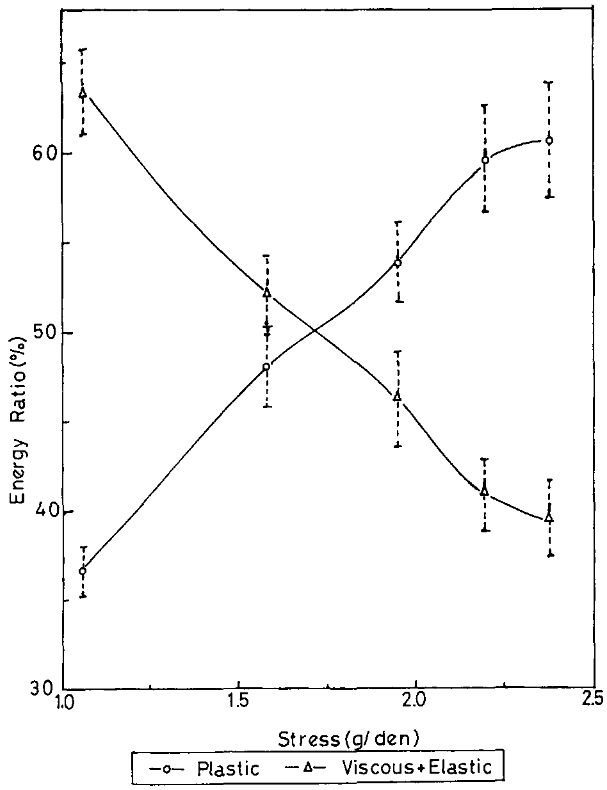

Figure 5 Plot of plastic vs. viscous + elastic components (strain rate is 20% per min).

certain interesting features come to light. At any given strain rate, the various energy components, viz., viscous, plastic, and elastic components, show an increasing trend (Table II). But for the percentage contribution of each individual component to the total energy when plotted for different strain levels (at the same strain rate), the viscous and elastic energy components decrease while the plastic energy component increases with increasing strain levels (Fig. 4). This is understandable, as without an increase in the plasticity of the system, failure cannot occur. The resistance to failure comes from the viscous and elastic components. Their contribution slowly decreases with increasing strain level, indicating the changeover in the material behavior, which is predominantly elastic and viscous at the initial stages to the plastic behavior at later stages, leading to failure. A plot of the plastic and viscous + elastic (V + E), against stress indicates the crossover point which should theoretically give the stress beyond which there would be an irreversible plastic flow leading to material failure in creep experiments (Fig. 5).

When the percentage contribution of each component to the total energy is plotted for different strain rates at a particular strain level, an interesting feature is observed (Fig. 6): While the plastic and elastic energy components show a decreasing trend, the viscous component increases with increasing strain rate. Unlike the stress–strain curves, there seems to be clear evidence of the effect of the strain rate on the viscous component. It is the only component that shows an increase in its percent contribution to the total energy as against the elastic and plastic components. As the viscous component is known to be the time-dependent part of the total

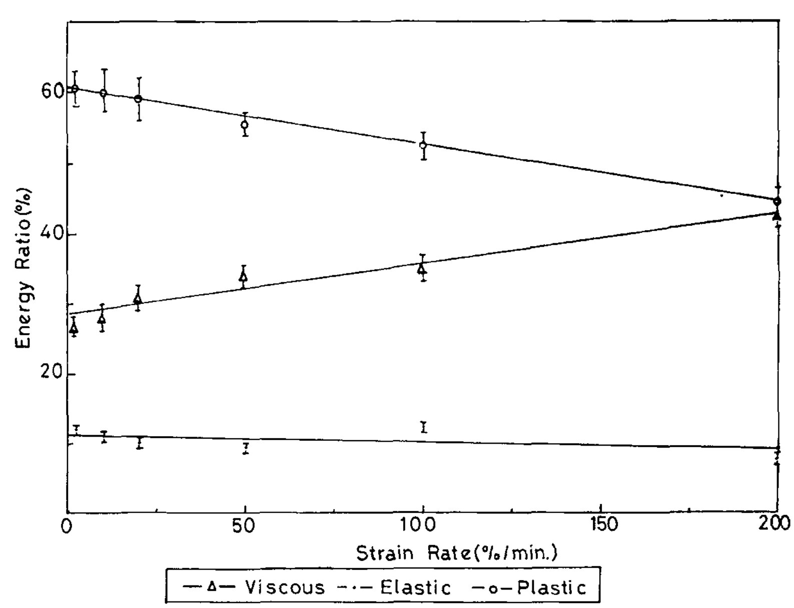

Figure 6 Effect of strain rate on viscoelastic components (at 12% strain level).

energy, any change in the strain rate should reflect on the extent of the contribution of this component.

CONCLUSION

Thus, it is evident from the above that quantification of different components of the viscoelastic response would throw more light on the material properties than will simple tensile tests and should prove to be a useful methodology in assessing any chemical modification.

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