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SHORT COMMUNICATION

CREEP AND LOW STRENGTH OF SPIDER DRAGLINE SUBJECTED TO CONSTANT LOADS

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ABSTRACT. Major ampullate (dragline) silk is attracting significant attention as a potentially useful engineering fiber. This interest is motivated by reports that the silk exhibits high mean strength, stiffness and toughness as measured in tensile tests. However, the typical testing conditions (constant strain rate; experiment completed within less than an hour) imposed during such assessments do not reflect typical demands (e.g. ability to support constant load for long times) made on real high-tensile materials. We demonstrate here that *Nephila clavipes* major ampullate silk subjected to constant loads performs poorly: its breaking strength is significantly lower than that measured in conventional constant strain rate tests, and even very small constant loads can cause elongation to increase appreciably over long timescales.

Keywords: Creep, dragline, *Nephila clavipes*, silk, strength

There is much current interest in major ampullate (dragline) silk, inspired by its headline strength, stiffness and toughness (Viney 2000b; Lazaris et al. 2002; Kubik 2002). Attempts are being made to produce economic quantities of silk-like protein in genetically altered organisms (Scheller et al. 2001; Lazaris et al. 2002), for spinning into high performance fibers. However, a functional engineering material must maintain its dependable properties, in an appropriate environment, for a serviceable period of time. The long-term tensile durability of spider silks has received scant evaluation. In this communication, we demonstrate that silk can undergo creep and catastrophic failure under ambient conditions, at stresses far lower than those needed to cause yield or fracture in a conventional (constant strain rate) tensile test.

The tests that are routinely used to characterize the tensile properties of silk and other fibers are performed at a uniform strain (elongation) rate (Viney 2000b), of the order of 10^{-4} s^{-1}. In other words, the specimen is forced to extend by 0.01% of its original length every second until it breaks (which typically occurs within 30 minutes of starting the test). The changing force needed to achieve the constant strain rate is recorded, and the force–elongation data are commonly re-scaled and presented in the form of a stress–strain plot. Here stress is the force applied parallel to the length of the fibre, divided by the original cross-sectional area of the fibre; strain is the resultant difference between the present and original lengths of the fibre, divided by the original length of the fibre. Such plots are conventionally used to define the yield stress or yield strength of the material, where the behavior deviates significantly from the initially linear relationship between stress and strain. If the test is interrupted and the load is removed before the yield stress is reached, the sample returns to its original length almost immediately, i.e. the sample behaves in a predominantly elastic manner. If the test is continued until the yield stress is exceeded, subsequent removal of the load does not result in immediate recovery of the original length; indeed, the original length may never be recovered. Stress-strain plots are also used conventionally to define the fracture stress or fracture strength of the fibre, in other words the stress at which the fibre breaks.

However, there are few instances where fibers would actually be subjected to a constant strain rate while in use. A more practical application might involve fibers being required to maintain a given tension, or carry a particular load, over an extended period of time. Under such circumstances, it becomes necessary to consider the time-dependence of the stress-strain behavior. Fibers subjected to a fixed load (and therefore fixed stress) for long times can respond with a gradual, continuous increase in strain until they break; even if the stress is signifi-

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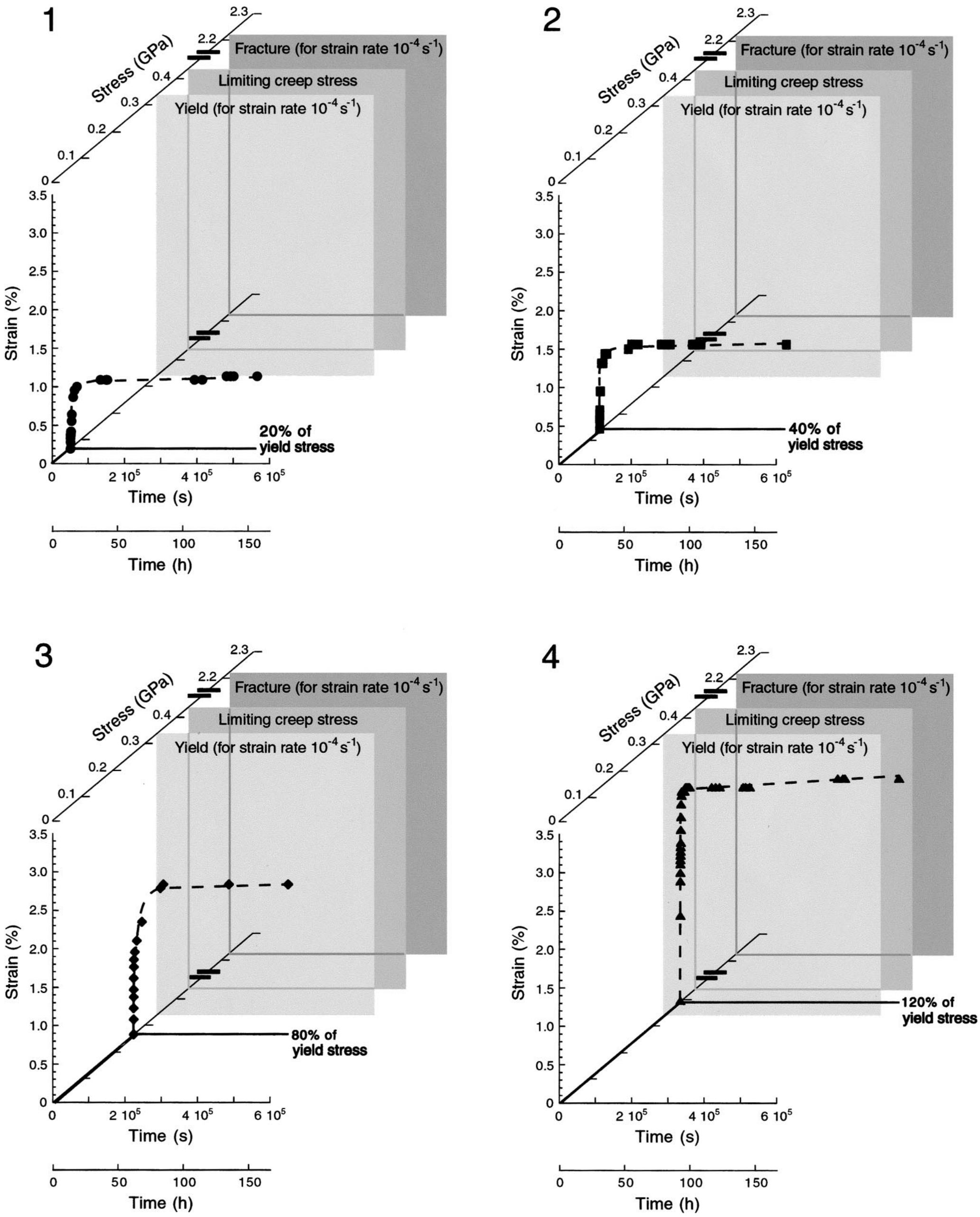

Figures 1–4.—Creep behavior of *N. clavipes* dragline tested in air under ambient conditions. In each case, plot symbols and dashed lines show creep (strain vs time), and a solid line shows the corresponding stress history (stress vs time). Each experiment was performed over a period of at least 100 hours. Shaded planes mark the yield stress (measured at a strain rate of 10^{-4} s^{-1}) and the breaking stress (measured at a strain rate of 10^{-4} s^{-1}), and also draw attention to the existence of a limiting creep stress as defined in the text. 1. Applied stress is 20% of yield stress. 2. Applied stress is 40% of yield stress. 3. Applied stress is 80% of yield stress. 4. Applied stress is 120% of yield stress.

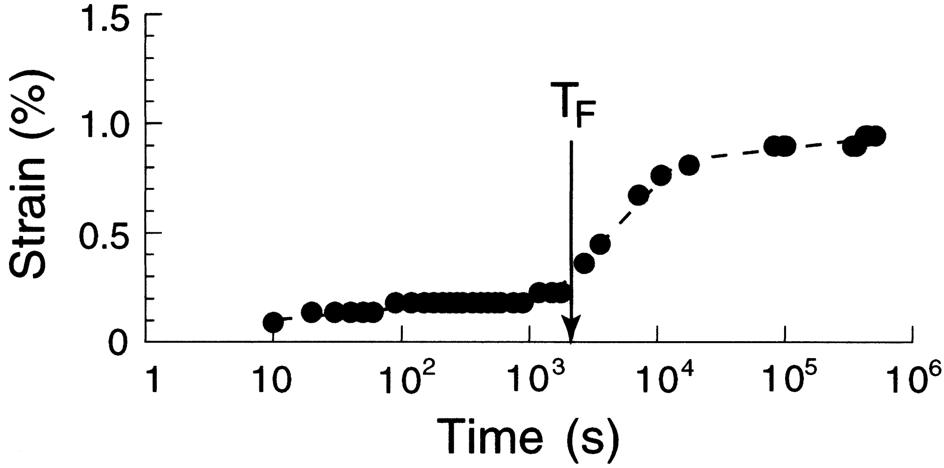

Figure 5.—Creep curve for sample loaded to 20% of yield stress (Fig. 1), re-scaled on a logarithmic time axis so that the initial stages of creep are emphasised. T_F is the approximate duration of conventional tensile tests performed on spider dragline at a constant strain rate of 10^{-4} s^{-1}; such tests are typically completed (the fiber fails by breaking) in less time than it takes for creep to make its most significant contribution.

cantly less than the breaking stress or even the yield stress as defined above. This deformation to failure at constant stress is referred to as creep. We have previously shown (Bell et al. 2002) that the tension due to supercontraction (Work 1981) decreases rapidly in restrained dragline when it is wet, with the implication that wet silk will also undergo significant creep under constant load. The results presented below explicitly reveal an even stricter limitation on the possible applications of silk and its biomimetic analogues: creep can occur even at ambient humidity, and even if the applied stress is small.

Nephila clavipes (Linnaeus 1767) (Araneae, Tetragnathidae) spiders were provided by Angela Choate, University of Florida, Gainesville, FL; identification was confirmed by Dr Scott Stockwell at the US Army Research Development and Engineering Center (Mello et al. 1995). Major ampullate silk was reeled (Work & Emerson 1982; Thiel et al. 1994) from spiders at 1 cm s^{-1}, which corresponds approximately to the rate at which the silk is spun during web construction. Spiders were not anesthetized. Silk was stored on cardboard supports (Carmichael & Viney 1999) and kept in sealed containers in the dark until needed for testing. The silk retains its molecular organization, and therefore its physical properties, indefinitely when stored under these conditions. We used conventional constant strain rate tests, performed at 10^{-4} s^{-1}, to determine the average yield stress and fracture stress of the silk. To quantify the creep characteristics, we then set up a series of experiments in which a small load, a block of an appropriate number of staples, was carefully suspended from a length of single-filament fiber inside a vertical glass tube that provided a shield against draughts. The initial length (gauge length) of the fiber samples was approximately 20 cm; this is an order of magnitude longer than the gauge length which is used in constant strain rate tests (Viney 2000b), but is necessary here in order to provide readily measurable extensions during creep. The weight of the staples was chosen to apply a set percentage of the yield stress to the fiber. (Although we are interested in conditions that cause the fiber to break, it is conventional to quantify creep stress in relation to yield stress.) The staples were attached to the fibre with a small dab of cyanoacrylate superglue. At the start of each experiment the silk sample was straight but not under load, with the staples resting on a support that could be withdrawn smoothly and quickly through the lower end of the glass tube. A combination of millimeter-grid graph paper attached to the glass tube and a vernier cathetometer standing on the benchtop allowed sample extension to be monitored as a function of time. Ambient conditions were 19 ± 2 °C and 60 ± 5% relative humidity. Creep experiments were performed three times at each value of applied stress, to take account of the intrinsic variability of dragline tensile properties (Pérez-Rigueiro et al. 2001); in each case data from the sample that survived for the median lifetime are displayed in Figs. 1–4.

Two notable observations emerge from this study. First, we can define a limiting creep stress: if samples are loaded smoothly and quickly to a constant stress lying above the limiting creep stress, they break within a few seconds of the stress being applied. The magnitude of the limiting creep stress is equal to approximately one fifth of the fracture stress that we recorded in conventional constant strain rate tests. Secondly, creep is significant at stresses that are small compared to the conventional yield strength. This behavior becomes especially apparent over timescales that are not accessed in typical constant strain rate tests, as emphasised in Fig. 5.

We chose *N. clavipes* dragline for our study because this material has been used widely in several laboratories where the mechanical properties of spider silk are characterized; albeit at constant strain rate rather than our present condition of constant load. Whether our observations of its creep behavior are broadly representative of spider dragline in general remains to be explored, given that similar silks from different species can exhibit markedly different mechanical properties (Viney 2000a). The effects of temperature and humidity on creep behavior also require further study. Conditions recorded in the present work are representative of those within the daily range experienced by *N. clavipes*, at any time of year, in its native habitat in Gainesville, Florida (US National Climatic Data Center, www.ncdc.noaa.gov).

Our observations expose limitations of unmodified spider dragline silk (and any fully biomimetic analogues) for applications in which high unidirectional loads must be supported for long times without failure or continuously increasing deflection. The fact that native dragline is made up of molecularly interconnected crystalline and amorphous zones in series (Viney 2000b) is consistent with the propensity of the material to creep. A similar microstructural susceptibility to creep is exhibited by so-called ''ultrastrong'' polyethylene (USPE) fibers such as Spectra (Prevorsek 1995). This study highlights a need for caution when transferring lessons from Nature to technology. Silks that are designed for a natural in-service lifetime of minutes (e.g. major ampullate silk used as dragline) or days (e.g. major ampullate silk used as web frame silk) should not be expected to necessarily exhibit properties that will be retained over timescales relevant to high-tensile engineering materials.

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