From Carbon Nanotube Yarns to Sensors: Recent Findings and Challenges

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Abstract: Carbon nanotube (CNT) arrays can be drawn into a web and then twisted into threads. These CNT threads contain thousands of carbon nanotubes in their cross-section and can be further composed into yarns consisting of one or more threads. CNT yarns exhibit significant mechanical stiffness and strength and low electrical resistivity. More importantly, CNT yarns exhibit piezoresistance that could be used for sensing purposes. In order to use carbon nanotube yarns as piezoresistance-based sensors for structural health monitoring, it is necessary to determine the change in resistance of the CNT yarn as a function of its mechanical strain or stress. This paper presents a succinct summary of the piezoresistive response of CNT yarns and the effect of the strain rate, strain level, mechanical properties, the geometry and lateral constraint. Strain rates affect the strength and failure mechanisms of CNT yarns, and their electrical properties. High strain rates show increased tensile strength and a positive piezoresistivity while low strain rates favor a higher strain to failure and a negative piezoresistivity. However, the sensitivity of the free CNT yarn is relatively unchanged with varying strain rate but strongly dependent on the strain level and its geometry. The lateral constraint occurring when CNT yarns are integrated in polymers or composite media most certainly affects their piezoresistive response.

Key-Words: - Carbon Nanotube Yarn; Piezoresistive Sensor; Mechanical Response; Electrical Response; Parametric Effects; Phenomenology.

1 Introduction

Carbon Nanotubes (CNTs) have been receiving increased consideration for structural health monitoring due to their multifunctional properties. Single wall carbon nanotubes (SWNTs), one-atomthick layers of graphene sheet wrapped into cylindrical tubes, have a diameter of approximately one nanometer. It is impossible to handle these structures in this scale without microscopic aid presently. To scale up its size, CNTs are spun into yarns; axially aligned CNT bundles that are a few microns in diameter. Their macroscopic scale permits their utilization in structural components and their tailorable aspect ratio provides a good fit for sensing in composite structures. Furthermore, they have a unique sensitivity to mechanical strain.

The aim of this study is to determine the effect of quasi-static strain rate, strain level, the mechanical properties and geometry of the CNT yarns and the effect of lateral constraint on their piezoresistivity. Strain rates affect the strength and failure mechanisms of CNT yarns, and their electrical properties. High strain rates lead to increased tensile strength and a positive piezoresistivity while low strain rates favor a higher strain to failure and a negative piezoresistivity. However, the sensitivity of the free CNT yarn is relatively unchanged with varying strain rate but strongly dependent on the strain level and its geometry.

2 Problem Formulation

The CNT yarns used in this study were grown from a CNT array on a substrate and spun from a vertically aligned CNT with no post-processing. A Si wafer with alumina (Al₂O₃) buffer layer was used with an iron-based catalyst both magnetron sputtered. The as-spun CNT array was approximately 400 µm in height, with a distribution of 1 up to 6 or 7 walls. The yarn's diameter was about 25-30 µm and the angle of twist was about 30°. Densification was achieved with acetone. The free CNT varn used for the tests is shown in Fig. 1a. Figure 1b, shows the CNT yarns integrated in a polymeric medium using a silicon rubber mold while Figure 1c shows the experimental set-up of the free yarn in a testing machine.







Fig. 1 - (a) SEM image of CNT yarn used for experiments (image taken using JEOL JSM-7100FA FE SEM). (b) Optical image of the constrained CNT yarn sample in a polymeric beam with strain gauge

mounted on it. (c) Optical image of experimental set-up to characterize the CNT yarn sample.

2.1 Free or Unconstrained CNT Yarn

A mechanical testing machine was used to perform the mechanical tests while an Inductance-Capacitance-Resistance (LCR) reader measured the electrical response. A Mechanical Testing System (MTS) Criterion 43 system was controlled via the TestWorks4 software and was programmed to apply a uniaxial tension load to the sample at several displacement rates. The MTS MultiCycle package was used to apply both cyclic loading and load up to failure. The load applied to the sample and the cross-head extensions were recorded as a function of time. Additionally. the LCR reader simultaneously measured changes in resistance during the tests. Once the maximum load was reached, the loading machine stopped and then gradually unloaded the sample until the crosshead extension reached its original position. The results were analyzed using MATLABTM and Microsoft ExcelTM. The LCR was connected to the CNT yarn sample through wire clips attached at both ends (Fig. 1c).

2.1.1 Strain Rate Effect

A cyclic loading test was performed up to 3 cycles to determine the piezoresistive response of the CNT yarns under variable quasi-static strain rates. Fig. 2 shows the comparison of the results obtained at two different strain rates. At a strain rate of 0.006 min^{-1} , the resistance increased during the loading segment and decreased as the load was released. Upon application of the load, there would be a reduction in the contact length of the CNTs in the yarn causing a tunneling distance across charge carriers. This separation of CNTs and their bundles leads to an increase in resistance. At a very low strain rate, 0.001 min⁻¹, a negative change in resistance was observed. As seen in Fig. 2, this negative relative resistance change was observed in both the loading and unloading segments of the test. The negative piezoresistivity could be explained due to the time factor associated with low strain rates. The long time associated with the slow strain rate for the load transmission to reach the critical length for normalized strain under low strain rate conditions is enough for the CNT yarns to experience relaxation. The relaxation is accompanied by rearrangement of CNTs networks due to the increased possibility of sliding between the overlapping CNTs and the bundles producing new surface areas.

It could be noted that the resistance changes at the lower strain rate (0.001 min^{-1}) do not return to the origin or initial point for subsequent cycles unlike at the higher strain rate (0.006 min^{-1}) . This could be explained by the inability of the CNT yarn to recover fully due to slip at low strain rates.

It is proposed that two phenomena govern the electrical response of the CNT yarns:

- (1) An increase in resistance due to an elastic expansion of the carbon nanotubes in their bundles attributed to a decrease in contact length during the loading segments and a corresponding decrease in resistance due to increase in contact area by the contraction of the carbon nanotubes in the bundles during the unloading segments; and
- (2) A decrease in resistance due to intertube/inter-bundle slippage (inelastic shear motion) caused by yarn's relaxation and structural reformation during the loading segments, and a continuous decrease in resistance during unloading as the yarn recovers its (conductive) structure.

In the case of the higher strain rates, the first phenomenon dominates during both the loading and unloading segments. At very low strain rates, the second phenomenon dominates the loading segment and since this action is irrecoverable, any increase in resistance during the unloading segment is not expected or feasible. The computed gauge factors for a variety of strain rates tested are displayed in Table 1.



Fig. 2 – Relative resistance change-strain curves at a 1 %-maximum strain for strain rates of 0.001 min⁻¹ and 0.006 min⁻¹, respectively.

Table 1 – Gauge factors of CNT yarns at various strain levels.

Strain Rate (min ⁻¹)	0.0006	0.006	0.06	0.6	6
Gauge Factor	0.15	0.16	0.16	0.18	0.20

2.1.2 Strain Level Effect

To study the effect of increasing strain level on the CNT yarn, tests were conducted at maximum strains of 0.1 %, 0.5 %, and 1.0 % at a strain rate of 0.006 \min^{-1} . Six samples were tested at 0.1 %-maximum strain. At 0.1%-strain level in a cyclic loading, the stress-strain curve corresponding to cycle 1 is shown in Fig. 3a1. There is no defined hysteresis rather a significant amount of noise from the equipment. The absence of hysteresis is quite expected since there is very low stress built up in the sample at a very low strain. At a maximum strain of 0.1 %, the CNT yarn undergoes very small strains, which are not large enough to cause any significant development of plastic deformation. For the samples tested to a maximum strain of 1.5 % and 2.5 %, the relative resistance change was much larger as seen in Fig. 3b,c, with the gauge factor reaching a value of 0.45. At higher maximum strain levels, CNT yarns tended to exhibit higher sensitivity as expected. It was observed from Fig. 3 that the relative resistance change was higher for the 1.5 %strain compared to that of 2.5 %-strain. This suggests that there may be an additional factor contributing to the CNT yarn's piezoresistivity other than the strain level. Although the strain level (1.5 %) was lower than at 2.5 %, the stress built up in the sample during the test was higher, 232 MPa, amounting to a surplus of about 32 MPa. To digress this discrepancy, the relative resistance changestrain curves are presented alongside the relative resistance change-stress curves in Fig. 4. At higher strains, due to the onset of plasticity, the stressstrain relationship becomes nonlinear, and the piezoresistivity comes from stress contributions rather than strain. It is quite visible from within the elastic region, 0.1 %-strain (Fig. 4a1,2) for example, that the corresponding relative resistance changestrain curve and the relative resistance-stress curve were identical. This is because the stress is directly proportional to the strain in this region. Beyond the elastic region (Fig. 4b), the strain may not account for the entire piezoresistive effect. Thus, the piezoresistivity of the CNT yarn should be correlated to the stress only.

The unloading curve of the piezoresistive hysteresis appears not to return to the origin and often times returns to a higher relative resistance value than that of the loading curve. This is the complete opposite of the observed unloading curve of the mechanical hysteresis, where the unloading path was below the loading curve and always returned to the origin for the first cycle. The analysis of the computed gauge factors for the strain levels tested shows that unlike the strain rate, the strain level itself accounts for the variance in gauge factor values (Table 2).





Fig. 3 – Piezoresistive responses of CNT yarns during first cycle at a strain rate of 0.006 min⁻¹: relative resistance change-strain curve for the following strains: (a1) 0.1 %; (a2) 1.5 %; (a3) 2.5 %.







Fig. 4 – First cycle curves at a strain rate of 0.006 \min^{-1} . (a1) Relative resistance change-strain curve of 0.1 %-strain. (a2) Corresponding relative resistance-stress curve. (b1) Relative resistance change-strain curve of 2.5 %-strain. (b2) Corresponding relative resistance-stress curve.

2.1.3 Geometrical Effects

The effect of geometrical parameters such as length and diameter of the CNT yarn on its piezoresistivity was also evaluated. Increasing the aspect ratio (length/diameter) of the CNT yarn, will increase the area available for frictional forces that develop between the CNTs. Consequently, a longer CNT yarn will experience increased friction. For CNT yarns with reduced diameter, the assumption is that piezoresistivity is proportional to stress and that stress is proportional to friction. However, it is important to note that twist contributes to stress transfer in ways dissimilar to fibers. Low twist means that the CNT yarn properties rely on the CNT yarn's dimensions: length and diameter.

CNT Yarn's Diameter: To study the impact of crosssectional area on the CNT yarn's resistance response, a simple assumption is made. It is assumed that the load that transfers from each CNT bundle inside the CNT yarn is proportional to the contact area while the stress level in the bundle into which the force has been transmitted is equal to the force divided by the cross-sectional area. Also, considering the relationship between resistance, R, and cross-sectional area, A, an increased crosssectional area (diameter) would amount to a decrease in the resistance for a constant resistivity. CNT yarns of different diameters, 25-29 μ m and 47-50 μ m, were used in this study. Five CNT yarn samples with a length of 25 mm were prepared and tested to failure maintaining the same testing parameters as in prior sections.

Although the elastic modulus and tensile strength are higher for the CNT yarn of smaller diameter, CNT yarn A (43-50 μ m-diameter) exhibits a higher resistance change than CNT yarn B (25-30 μ m-diameter) as observed in Fig. 5. It is clear from Fig. 5b that more load was transferred to CNT yarn B but the corresponding resistance change did not clearly show it. This may indicate that the previous notion of an increased cross-sectional area resulting in a decrease in resistance change does not hold for a CNT yarn due to its resistance being accounted for by mostly contact resistance.





Fig. 5 – Effect of CNT yarn's diameter on its piezoresistive response. (a) Relative resistance change-strain curve at 0.006 min-1 to failure. (b) Corresponding stress-strain curve.

The CNT yarn with high diameter, due to the higher CNT volume, experiences more friction in tension and as such, exhibits a better stress-toresistance transmission mechanism. This may not be the case for fibers where covalent carbon bonding outweigh contact forces, hereby accounting for nearly all of the stress.

CNT Yarn's Length: Another set of CNT yarn samples with three different gauge lengths, 10 mm, 15 mm and 25 mm, were prepared to compare the effects of varying length on the piezoresistivity of the CNT yarn. The CNT yarn may exhibit a higher tensile strength with decreasing gauge length, and similarly, a lower tensile strength with an increased length, the same way as any material with random defects would be weaker with increasing length. From the relative resistance change curve of Fig. 6, it is observed that the longer the CNT yarn, the greater the resistance change. The 15 mm-long CNT varn sample exhibited a similar response to that of 10 mm-length. However, the difference became very pronounced when the relative resistance readings were compared with those of the 25 mmlong CNT yarn sample. This could be explained by the additional contact area the longer CNT yarn provide for charge carriers to either separate or come in contact during the tests. For each of the gauge lengths tested, it was determined the displacement rate transition at which the piezoresistivity sign would reverse. The results are shown in Table 2. The 10 mm- and 15 mm-long CNT yarns have a lower transition displacement rates than the 25 mm-long CNT yarn. This could be explained by the limited effect of sliding in higher gauge lengths due to increased contact lengths. The interfacial contact means that stress redistribution is engaged more uniformly than at shorter gauge lengths, which provide less contact area. This is demonstrated by a higher sensitivity of the 25 mmlong CNT yarn, with a higher gauge factor of 0.4, compared to 0.13 of the 15 mm-long CNT yarn and 0.07 of the 10 mm-long CNT yarn.



Fig. 6 – Relative resistance change-strain and stressstrain curves at 0.006 min⁻¹ to 1 %-strain.

Table 2 – Piezoresistivity sign of CNT yarns in terms of displacement rate and gauge length.

Disp. Rate (µm- min ⁻¹)	25	50- 75	150- 300	500- 750	1000 and above
Length					
(mm)					
10	(-)	(-)	(-)	(+)	(+)
15	(-)	(-)	(-)	(+)	(+)
25	(-)	(-)	(+)	(+)	(+)

(-) = negative; (+) = positive

2.2 Constrained CNT Yarn

If CNT yarns are to be used as integrated sensors, their piezoresistive response needs to be determined when inside a medium, polymeric or composite. The piezoresistive response of the CNT yarns integrated in polymers is presented next. It is worth

mentioning that the strain is obtained directly from the bonded strain gauges, which may be different from that determined from the applied load using a beam theory formulation. The strength of CNT yarn can vary across the radial length, so the tests for each section was performed from the CNT yarn cut from the same section and batch of the CNT yarn used previously. The experimental setup allows pure bending in the central section of the simply supported rectangular polymeric beam with supports at points A and B (Fig. 7). A 30 kN load cell connected from the MTS machine is used to apply a load, P/2, at points C and D, which are spaced equidistantly from the end and middle of the beam. Two strain gauges are bonded at the center of each the top and bottom surfaces of the beam. The strain gauge at the top measures compressive strain, \mathcal{E}_1 , from bending and conversely, the strain gauge at the bottom measures the tensile strain, \mathcal{E}_2 . Thus, pure bending occurs between the loading points, C to D, where there is a constant bending moment of M=P*d/2 with the radius of curvature ρ . A relation between the M and ρ can be used to compute the stress and strain and compare the latter to the values from the strain gauges.

Since the CNT yarn is embedded on one side alone, close to the surface, it can be assumed that the strain measured by the strain gauge is the one experienced by the CNT yarn. Polymers are homogeneous materials, and thus the strains on the top and bottom surfaces should be identical. However, the side of the polymeric beam with the CNT yarn may experience higher strains due to cracks and cavities created by its integration.



Fig. $\overline{7}$ – Schematic of four-point bending of the sample with two pieces of strain gauge glued to both sides.

2.2.1 Strain Rate Effect

To measure the impact of strain rate as reported previously [1], the results obtained at different strain rates, 0.0005 min⁻¹, 0.003 min⁻¹ and 0.006 min⁻¹, were graphed (Fig. 8). At the very low strain rate of 0.0005 min^{-1} and below, the rate sensitivity effect is not conspicuous in a constrained CNT yarn as it was the case in free CNT yarns. Due to the very significant disparity between the results from the constrained at 0.006 min⁻¹ to both 0.0005 min⁻¹ and 0.003 min⁻¹, the strain level was kept at 0.05 min⁻¹. A noticeable nonlinearity was observed as the strain rate is reduced. The nonlinearity also affects the values of the gauge factors as any linear trend imposed on graph will most likely consider peak and valley points that are a result of the amplification of background noise at low strain rates. There is no observable negative piezoresistivity at the low quasi-static strain rates. It is observed that the gauge factors increased significantly when the strain rate reached a value of 0.006 min^{-1} . At 0.006 min⁻¹, the obtained gauge factor was approximately 23.0 in comparison to the values obtained at 0.0005 min⁻¹ and 0.003 min⁻¹ which were 19.0 and 1.3 respectively. With an increase in strain rate, the linearity of the response improved. It is worth mentioning that a linear response is imperative for practical sensing applications of these piezoresistive CNT yarns.

These results could be explained by the limitation of slippage encountered in a constrained medium. In a free or unconstrained state, the CNT varn under low strain rate loading experiences slipping, causing the CNTs to rearrange their structure simultaneously with the loading. Depending on how low the rate of loading, the fiber rearrangement can either equalize the rate of deformation leading to a weak load transfer mechanism and mild slippage or it might outweigh the loading rate leading to a high slippage effect. Slippage accounts for negative piezoresistivity in CNT yarns [1].



Fig. 8 – Relative resistance change-strain curves of constrained CNT yarns under tension at varying strain rates: 0.0005 min⁻¹, 0.003 min⁻¹ and 0.006 min⁻¹, respectively.

2.2.2 Tension versus Compression

The constraint of the CNT yarn prevents the unbundling or fiber unraveling which is typically observed in a free or unconstrained CNT yarn. Also, loosening of twists and fiber sliding associated with changes in geometrical properties of the CNT yarn is limited. Thus, the polymer keeps the CNT yarn in place and intact during tensile deformation, providing more surface area for load bearing.

Fig. 9a shows the piezoresistive responses of a sample under tension. At 0.1 %-strain, a gauge factor of approximately 29.3 was obtained. This is a conformation of the expected response but also incredibly high for a low strain and much higher than metallic foil strain gauges. The obtained gauge factors for unconstrained CNT yarns from the same batch of CNT yarn was lower than 1. This also validates the postulated effect of slippage and fiber disintegration of CNT yarns under tension. Figure 9b shows the piezoresistive response of the constrained CNT yarn under compression for 0.1 %strain at a strain rate of 0.006 min⁻¹. The corresponding value obtained for the gauge factor (~ 21.2) is significantly lower than that in tension (~ 29.3). The increase in contact area due to an increased fiber volume fraction under compression will result in a significant decrease in resistance change relative to stress in the medium. Also, since contact resistance accounts mostly for the piezoresistivity of the CNT yarn, the frictional motion is restricted under compression. The high

gauge factor signifies that the CNT yarn with its weak carbon to carbon bond due to CNTs not spanning the sample length, experiences change in resistance mostly from contact resistance or contact area. However, it is assumed that inclusion of the fiber affects the homogeneity of the polymer as they may become a source for microvoids that may coalesce during testing. Thus applied load to achieve same strain may vary across sample for both tension and compression.



Fig. 9 – Relative resistance change-strain curve of constrained CNT yarn sample up to a maximum strain of 0.1 % strain at a strain rate of 0.006 min⁻¹ under: (a) tension; (b) compression.

2.2.3 Effect of Lateral Constraint

There is a clear difference in piezoresistivity between the constrained and unconstrained CNT yarns [2]. For the samples tested at 0.003 min⁻¹ to a maximum strain of 0.1 %, the gauge factors of the varns constrained and unconstrained are approximately 14.8 and 0.1, respectively. From Fig. 10b, a gauge factor of 30.7 was recorded for the constrained CNT yarn against 0.2 for the free CNT varn when tested up to a maximum of 0.1 % strain at 0.006 min⁻¹. It could be seen that the gauge factor increased from the free to the constrained but much significantly for the constrained CNT yarn at higher strain. For both conditions, when the strain rate was doubly increased, the value of the gauge factor was twice as found in lower rate. However, the strain rate does not affect the value of the gauge factor as much in a free CNT yarn when compared to a constrained yarn [3]. The curve of the constrained CNT yarn is also much linear and would produce higher gauge factors than that of the free CNT yarn. Consequently, the piezoresistivity of the constrained CNT yarn is higher than that of the unconstrained CNT yarn. Due to the discontinuous nature of the CNTs in a yarn, most of the yarn's resistance is produced by the contact resistance of the individual fibers. It is assumed that the fiber volume fraction increases due to the constraint imposed on the CNT yarn by the polymer medium. Thus, there is an increased fiber density. Furthermore, the constraint negates the impact of slippage, thus enhancing interfiber load transfer. A more uniform load distribution in the CNT yarn would account for the higher piezoresistive response. Table 3 presents the gauge factors for different strains levels at 0.003 min⁻¹ and 0.006 min⁻¹ strain rates.





Fig. 10 – Effect of lateral constraint on the piezoresistive response of free and constrained CNT yarns: relative resistance change-strain curves: (a) 0.1 %-strain (0.003 min⁻¹); (b) 0.1 %-strain (0.006 min⁻¹).

Table 3 – Average sensitivity (strain gauge factor) of three constrained and free CNT yarns in terms of strain rate (min⁻¹).

Strain	Strain	Gauge Factor	Gauge
Rate	Level	(Constrained	Factor
(\min^{-1})		CNT Yarn)	(Free CNT
			Yarn)
0.006	0.05	22.02	0.12
0.003	0.05	13.48	0.11
0.006	0.1	30.74	0.20
0.003	0.1	15.16	0.16

3 Conclusion

The piezoresistive response of laterally constrained CNT yarns was determined for the first time. The rationale of this study was to mimic the piezoresistive response of CNT yarn sensors that are integrated in polymers or composites. The response of unconstrained CNT yarns under tension had been determined previously and the results could now be compared with those in this study. All these results were obtained using quasi-static strain rates. It is very important to note that the previous results had shown that the strain rate plays a very significant role not only on the amount of piezoresistivity in the free yarn but also in its response varying from quasi-parabolic to linear. By subjecting a CNT yarn embedded into a polymer medium to four-point bending, the piezoresistive response of the constrained CNT yarns was determined under tension and compression. From previous research, the strain rate of 0.006 min⁻¹ is considered a high quasi-static rate which is the strain rate used in this study [1].

It is assumed that the distance between the carbon atoms are longer under tension. This leads to an increase in the resistance. At high strain rates, this phenomenon is dominant and consequently an increase in resistance is observed. At low strain rates, fiber slippage is high leading to a decrease in the resistance. Similarly, an increase in the resistance is also observed in the case of constrained CNT yarns under tension and this increase is even higher than that of unconstrained CNT yarns. Therefore, based on all previous results and those of this study, the following hypotheses are proposed. Because of the lateral effect exerted by the polymer host medium, the cross section of the CNT yarns cannot shrink but their length can increase along with the length of polymeric beam. Hence, the second phenomenon cannot occur when the CNT varn is embedded in polymeric beams. This leads to a significant increase in the resistance of the constrained CNT yarns. The relative resistancechange of both the constrained and unconstrained CNT yarn increases monotonically with the strain. However, the response of the constrained CNT varns is much more sensitive than that of the unconstrained CNT yarns. This difference between them may be explained by the lack of the effective slippage, fiber unraveling and subsequently, Poisson's effect of the unconstrained CNT yarn when inside the polymer. Higher sensitivity was observed for the samples tested at higher strain rates with gauge factors increasing with increasing strain.

The piezoresistive response of the constrained CNT yarns under compression was observed to exhibit a quasi-parabolic response. For both tension and compression, the relative change in resistance decreases slightly at first reaching a local minimum and tends to increase later. The hypotheses that were used to explain the phenomena of the CNT varn under tension could also be applied in the case of the constrained CNT yarns under compression. Increased fiber density means charge carriers becomes closer and the resistance decreases. Therefore, a higher gauge factor is obtained under tension than under compression. It is however difficult to compare the compressive response of CNT yarn in free and constrained states due to the premature buckling involved in testing a free yarn under compression. The negative piezoresistivity

experienced in a free CNT yarn in a low strain rate condition during loading was not observed at similar strain rate for the constrained CNT yarn. Therefore, a foregone conclusion is that slippage plays a deeper role in the resistance decrease upon loading at low strain rates.

The results in the present study constitute yet another step towards determining the complete picture of the piezoresistive response of carbon nanotube yarns. At this time, the authors are certain that the sensitivity of these yarns may be sufficient for integrated strain sensing and most certainly for damage detection. However, significant challenges remain in determining the effect of a large number of cycles on the piezoresistive response. Detailed experimental studies are being conducted to learn about the latter but also to determine the effect of twist and other structure, mechanical and electrical parameters on that piezoresistive response.

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