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Study of tensile behavior for high-performance fiber materials under high-temperature loads

Ayham Younes[1], Vignaesh Sankaran[1], André Seidel[1] and Chokri Cherif[2]

Abstract

Textile high-performance filament yarn subjected to extremely high thermal loads can be found in various technical application fields. Besides the mechanical loads, textile fiber materials have to also satisfy high safety requirements in these applications with respect to thermal loads. Some of the main fields of application in the field of mechanical engineering are turbines, drive devices, rocket components and fire protection coatings. Textile grid-like structures are also being increasingly used in civil engineering as reinforcements (textile concretes). The design and development of textile structures for these applications demands studying and acquiring the material behavior under high thermal loads. Neither sufficient data nor standardized testing methods have been extensively achieved for evaluating the tensile characteristics of filament yarns under thermal influences. Hence, studying the thermal behavior of these yarns, which are used as input material for the reinforcing structures, is essential. The impact of the standard atmospheric condition on the oxidation behavior of the yarns, as in the case of carbon filament yarns and their influence on the physicochemical and tensile mechanical properties, have to be studied as well. This paper aims to address this issue and provides an insight into the current research about the development and realization of a novel test stand and the subsequent study of tensile mechanical behavior for textile high-performance fiber material under extreme thermal loads together with their physicochemical behavior.

The application of high-performance filament yarns in technical as well as other application fields, such as fiber composite material for the automotive, aeronautics and aerospace industry, mechanical engineering and civil engineering, has increased manifolds. Continuing this trend for the future requires gaining a thorough knowledge about the material behavior under extreme load conditions. The study of applied material and its components under thermal loads (high temperatures) assumes huge significance in context with stringent fire safety requirements.

Textile materials with high-performance fibers made of glass and carbon having a very high strength potential because of their physical and chemical characteristics are used as reinforcements in various technical applications. As is the case in civil engineering, these materials are used for concrete reinforcements, which offer a decisive advantage over the existing conventional reinforced concrete technology for lightweight applications and architecturally demanding constructions. The suitability of textile reinforcing structures made of alkali resistant glass (AR-glass) and carbon filament yarn for the reinforcement of existing and new buildings under mainly resting loads has been

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extensively proven in initial stages of our research activities carried out under the German Special Research Field 528 "Textile Verstärkungen zur bautechnischen Verstärkung und Instandsetzung" (Textile Reinforcements for Structural Strengthening and Repair). In order to ensure the required structural solidification of the reinforcing textile grids made of yarns for on-site processing, as well as the activation of the characteristics potential of the textiles in the composites, these grids are applied with polymeric coating in the form of a watery dispersion on the basis of self-linked polymers (styrol butadiene (SBR)).[1–5] These applied textiles also have to satisfy very stringent safety standards amongst others with respect to structural fire protection.

Under the complex loading conditions, both the mechanical and the thermal loads influence the temperature-related structural changes in the reinforcing textiles. In order to understand the specific characteristics of the yarn behavior to high-temperature exposure, it is necessary to develop suitable test stands and testing methods for studying and analyzing the characteristics of the filament yarn under various test conditions. Thereby a sound knowledge on the temperature-related physical, chemical and tensile characteristics of the applied fiber material can be achieved and thereby subsequent inferences could be derived for critical thermal loads. Moreover, these also can form a fundamental work for the setting of an experimental characteristics database to characterize the temperature-dependent behavior of textile high-performance filament yarns and for the definition of safety criteria for textile-reinforced concrete under high thermal loads.

This paper initially presents a novel test stand developed based on the existing measuring techniques and test methods for determining the material thermal behavior, which has been adapted to the demands of textile yarn material. The experimental tests carried out using this novel test stand based on the standardized conditions conforming to the norm DIN 4102 for the fire behavior of components and building material and the specific temperature curve determined using various textile reinforcing layers during fire test at textile-reinforced reinforced concrete plates,[6,7] is then elucidated.

Initial situation and problem definition

Textile filament yarns show structural-related changes in their mechanical characteristics under thermal loads, which are, in most cases, accompanied by a decrease in their tensile characteristics. The existing data gathered in the field of fiber composite material so far are mostly based on their characteristic values, which are not directly determined at the yarn under a constant temperature load but measured through pre-heating and subsequent re-cooling during tensile tests.

Glass-fibers as stated in literatures undergo structural changes at temperatures of about 300–400°C, which are accompanied by a clear decrease of their bearing capacity.[8,9] Below the transitional temperature of 240°C, a clear increase in strains can be observed as shown in the viscosity analysis of glass.[10] The constant loading limit of the glass fibers under high temperature is given as 250°C,[11] although the first irreversible strength loss can already be noticed at temperature levels of below 200°C and with a short load period.[12] With further increasing temperatures, the tested E-glass filament yarns show a rapid decrease in strength, whereas the elasticity module remains unchanged.

Similarly, the high-temperature behavior of carbon fibers has also already been extensively researched. It is known that under a standard atmospheric condition, temperature in the range of 400–500°C leads to oxidation reactions in the fibers, during which carbon dioxide and carbon monoxide are discharged. This causes irreversible damage to the material, since the single filaments are destroyed and hence results in a clear decrease of the strength characteristics.[13–18] Further research also shows that even at partly lower temperatures of about 300°C initial oxidation reaction occur.[19,20] The temperature for initiating the oxidation process is dependent on influential factors, such as the carbon fiber pre-product (precursor) and process temperature during the fiber production.[21] These findings are also proven in other research,[22,23] which deal with the different types of carbon fiber with varying mechanical characteristics exhibiting differences in their oxidation behavior.

Furthermore, numerous scientific papers have studied the oxidation of carbon under the accelerating influence of catalyzers. In this case, it was recognized that elements or compounds such as alkaline metal, platinum, silver oxide and boron that function as catalyzers, can accelerate the oxidation reaction of carbon.[19,21,24,25] In contrast, carbon exhibits an extremely high thermal resistance under inert conditions when these oxidative influences are excluded. This is shown by the study on the mechanical behavior, the electrical conductivity and the thermal expansion coefficient using two carbon fiber types on the basis of polyacrylonitrile (PAN) and viscosity under high-temperature impacts in a vacuum.[26] From these studies, it could be inferred that the tensile strengths of carbon at temperatures about 1600°C remain nearly unchanged and the E-module decreases slightly at temperatures above 1000°C. Analog findings of unchanged strength characteristics of carbon until about 1000°C are also given in the study under inert gas atmospheres.[27]

The current experiments for the characterization of the temperature-related yarn mechanical properties are carried out on a pre-heated and subsequently

pre-cooled test specimen for determining its tensile strength. These methods only provide results concerning the residual strength of the yarn after being subjected to the thermal load. These do not reflect the real conditions where the actual behavior of the material has to be studied under the influence of an existing thermal load. Also, the cooling process of the pre-heated yarns might lead to molecular rearrangements as a result of re-solidification. Hence, the transfer of these results for the determination of the yarn tensile strength under simultaneous temperature loads is inaccurate and highly limited.

A special servo-mechanic testing machine with an integrated high-temperature chamber for ceramic composites is introduced by Ehlig et al.[7] The tests can be carried out both under room temperature conditions and vacuum/shielding gas. The test specimens are normally heated inductively and in the case of non-conductive material with the help of susceptors and heat radiation, where temperatures up to 1600°C are possible. The measurement of the strain is done with the help of a high-temperature extensometer integrated in the test chamber. This method is extensively used in the field of metallurgy and ceramics. Although technically feasible it is still complex to adapt this technique for the filament yarn, as the specimen clamping and strain measurements require intensive research. Also, a statistically reliable inference using this method requires innumerable trials.

Universal test machines for the tensile testing of metallic material under high-temperature loads are also commercially offered by the company Zwick GmbH & Co. KG. Here, the heating of the test specimen is carried out in an inductive oven, which is integrated to a tensile testing machine.[19] However, the test duration here is extremely time-consuming due to the required heating, stoppage and cooling time, besides the reproducibility.

The only existing test method in which single filaments are tested under simultaneous temperature loads is shown by Jesse.[2] Here, a test stand was developed, which allows the determination of the thermal extension coefficient, the elasticity module and the tensile strength of carbon filaments under high temperature and in vacuum. The electric conductivity of the carbon is used for the heating of the filament. Temperatures up to 2200°C can be achieved. The determination of the length changes is then carried out using an optical image analysis. A further proposed method also deal with development of a test stand for the characterization of carbon filaments with respect to the thermal extension coefficient in the longitudinal direction and radial to the cross-sectional level under temperatures of up to 2000°C in vacuum. The heating here is also inductive due to the conductivity of the carbon.[28]

However, given the fact that the carbon oxidizes under the influence of oxygen at high temperatures, these results are not transferrable for applications under room temperature conditions. Furthermore, the determined strength characteristic of a single filament need not wholly reflect the behavior of the filament yarn. Hence, the described test methods cannot be directly used for the evaluation of the high-temperature behavior of textile filament yarn and new suitable methods are required.

Design and development of a novel test stand

Based on the above scenario, a novel test stand was developed where the high-temperature behavior of the filament yarns can be studied for varying thermal load conditions under both standard and inert atmospheric conditions. A standard tensile testing machine Zwick Z100 used for tensile tests on textile high-performance filament yarn and also equipped with special clamping devices (rope sample supports) was modified and adapted to the new concept (Figure 1). A heat chamber fitted with an infrared radiator unit to simulate the temperature loading conditions was developed and integrated into the test machine. The measurement of the temperature and the regulation of the radiation were carried out using a thermal element, which is mounted directly in front of the test specimen (yarn) and connected to a controlling unit. The vertically clamped yarn traverses the heat chamber with a length of about 200 mm and is subjected to a specific test temperature. The temperature-dependent tensile characteristics, the temperature resistance under existing tensile loads and the remaining strength of pre-loaded yarns under thermal load can be determined. Test temperatures up to 700°C can be achieved and also variable temperature-time-curves can be applied.

In the newly developed test stand, the test specimen is enclosed in a sealed heat chamber. Hence, the alignment of the elongation measurement points is located directly outside the chamber. The effective testing length of the yarn is approximately 380 mm. The change in length of the yarn between the two measuring points is recorded for both the heated and normal sections outside the chamber.

The adaptation of the infrared heating principle has its own advantages over the existing inductive method. It offers a quick, flexible and reproducible option where the pre-heating and subsequent cooling times could be avoided. Also, the electrical property of the test specimen has no relevance, unlike in the inductive method. The infrared heating is an indirect heating method where the energy is transferred onto the material to be heated via electromagnetic radiation of
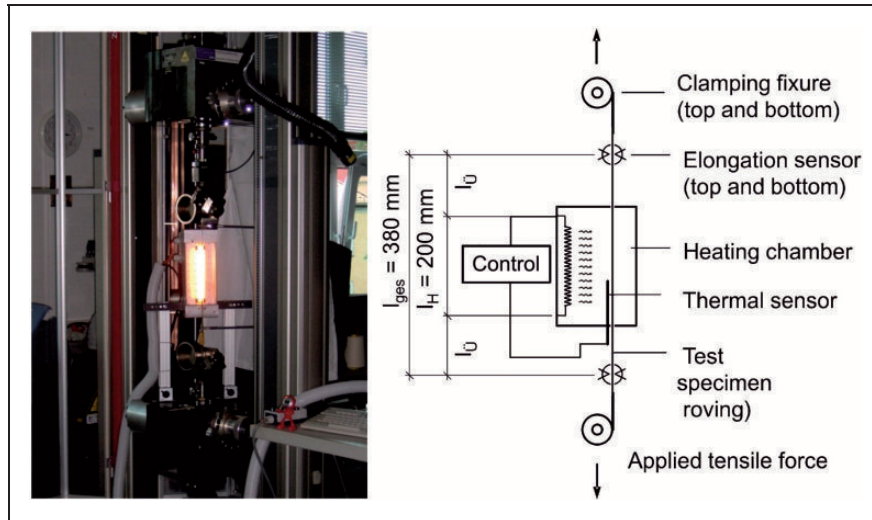
Figure 1. Novel test set-up to carry out strain tests under high temperatures and its schematic.

heating elements. The radiation spectrum of the electromagnetic wave in the infrared radiation starts above the visible light at about 0.75 μm and reaches up to the radio-waves with wave lengths of about 100 μm. Every test specimen is in a constant physical interaction with its surrounding because of the radiation. Outer radiation affects the specimen, which in turn itself dispenses radiation to its surrounding.

When the infrared radiation falls on the specimen, it is partly reflected, partly transmitted and absorbed by the surface, where the molecules are energized inside the material and the temperature rises. This process of reflection, transmission and absorption parts varies based on the type of material, surface consistence and wall thickness of the radiated body. It also depends on the impact angle and wave length of the dipping angle. As a consequence of these aforementioned aspects about the absorption behavior of textile filament yarn under infrared radiation, the temperature measured within the heating chamber according to Figure 1 directly at the thermo unit and that of the test specimen do not have to be necessarily identical. Since the electromagnetic waves of the thermo unit are absorbed and reflected differently by the carbon and glass filament yarns, a detailed analysis needs to be established. Hence, some measurements were first carried out here in the form of pre-tests, where a carbon fiber roving and a glass fiber roving were enwrapped with an additional thermo unit. The existing temperatures of these rovings could then be measured reliably with those two thermo units under infrared radiation (see Figure 2). It can clearly be seen that the temperature profile of carbon differs increasingly from the targeted temperature with increasing temperatures. The reason for this is that the electromagnetic waves are much more absorbed by the black carbon surface, which leads to an accelerated

heating of the carbon. Furthermore, it can be seen that starting at a temperature of 700–800°C, a rapid temperature increase can be found in the carbon, which continues to a little above 1000°C. The temperature then decreases again and evens out to the level of the control-temperature sensor. Using this effect, the thermal destruction of the carbon roving can be well observed due to the oxidation taking place and, thereby, the failure temperature under normal conditions could be identified.

In order to avoid the problems arising for a direct temperature measurement in the infrared area, the test yarn specimen is not directly exposed to radiation. The temperature is rather measured indirectly through a convective heat transfer from a metallic sheathing tube, which enwraps the yarns and is heated by the infrared radiator. This metal tube alignment in the heat chamber is located directly in front of the infrared radiator and absorbs a large part of the impacting infrared radiation to generate heat, which is then dispensed evenly and to all sides of the tube (inner and outer), as can be seen in Figure 3. In this way it is ensured that the yarn inside the metallic tube is completely exposed to the temperatures inside the tube, which, in turn, is in accordance with the test temperature.

As explained in the previous sections, the oxygen present in the room atmosphere determines the level of oxidation reaction under thermal loads for carbon filament yarn, which leads to a worsening of its tensile characteristics. The concentration and amount of oxygen influence the intensity of this reaction. In contrast, it can be taken that under inert conditions, the thermal stability of carbon is far greater than 2000°C, if no oxidative components are present in the carbon filament yarn.
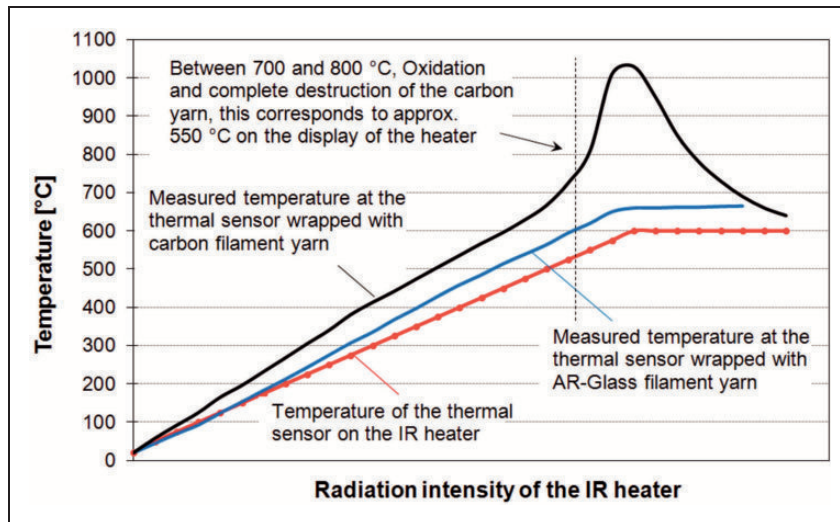
Figure 2. Temperature profile measured on the different material surfaces under infrared (IR) radiation.
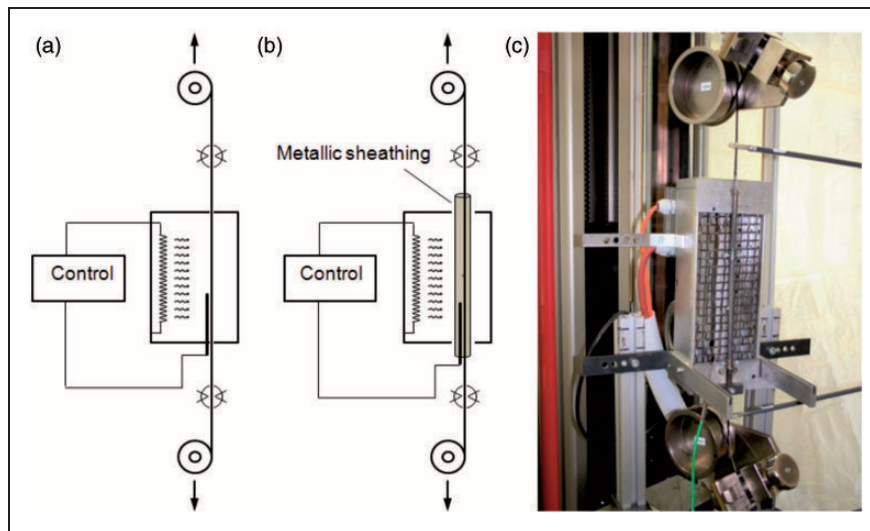

Figure 3. Modified test stand for the indirect infrared heating of the test body: (a) schematic of the initial test stand; (b) schematic of the modified test stand with metallic tube; (c) photo of the test stand.

In order to validate this experimentally and test the filament yarn under inert atmospheric condition, the test stand was suitably modified. Suitable provision was made inside the metallic tube so that an inert gas atmosphere condition could be created for the test yarn specimen. For this purpose, an additional optional connecting tube was used with the help of which the jacket tube could be flooded with pure Nitrogen (see Figure 4).

Thus, the new design of the test stand enables one to derive experimental results about the temperature dependence on the tensile characteristics of the yarn material both under standard and inert atmospheric conditions.

Experiments and results

Based on the novel test stand, an extensive experimental test program for the characterization of temperature-related material characteristics of high-performance filament yarns for AR-glass and carbon under various conditions was carried out. These are discussed in the following sections. An extensive test program for the characterization of temperature-related material characteristics of high-performance filament yarns for both AR-glass and carbon rovings (coated and uncoated) was also carried out and is summarized in Table 1. These yarns were first tested in their uncoated form as delivered in their original form and then with a coating
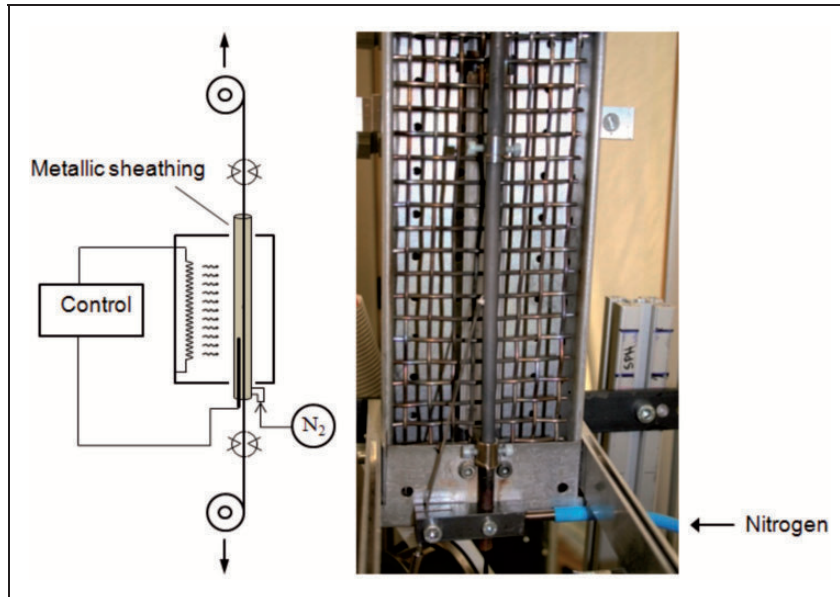
Figure 4. Modification for the temperature strain tests under inert (nitrogen) atmosphere and schematic.

Table 1. Specification of the applied material for high-temperature tests

Specification	AR-glass filament yarn (ARG)	Carbon filament yarn (CF)
Yarn count	640 tex	800 tex
Producer, type	Vetrotex (Cem-FIL)	Tenax®-E HTS 40
Density	2.75 g/cm³	1.79 g/cm³
Number of filaments	ca. 2000	ca. 12,000
Cross-sectional area	0.233 mm²	0.447 mm²
Filament diameter	12 μm	7 μm
Coating	Styrol butadiene (SBR)	
Coating content	8...10 Mass-%	

AR-glass: alkali resistant glass.

Table 2. Test plan for the determination of the yarn strength under thermal loads

Material	Test temperature $T_{Prüf}$	Heating duration
CF 800 tex – uncoated	20°C	[Reference tests]
CF 800 tex – SBR coated	100°C	8 min
	200°C	18 min
ARG 640 tex – uncoated	300°C	28 min
ARG 640 tex – SBR coated	400°C	38 min
	500°C	48 min
(10 tests each)	600°C	58 min
	700°C	68 min

SBR: styrol butadiene.

application made of SBS. The tests were carried out under oxygen-abundant room conditions. For the verification of the insights gained here, further experimental analyses on the temperature-dependent mass losses of the listed specimens were carried out with the help of which the destruction of the material was documented under the thermal and atmospheric conditions.

Tensile strength dependence on temperatures

The tensile strength dependence on temperatures test was carried out to determine the temperature-dependent tensile strength and stiffness of high-performance filament yarn made of AR-glass and carbon. Based on their behavior under a standard temperature of 20°C, different test series were carried out subsequently at gradually increasing temperatures of 100–700°C, with
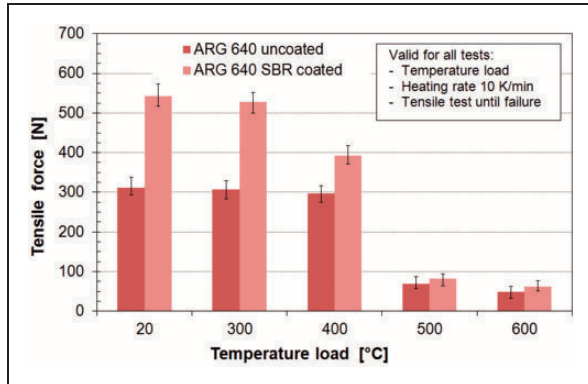
each series consisting of 10 tests each (see Table 2). All tests were carried out under standard room conditions, which means under oxygen-abundant atmosphere. Hence, it can be assumed that for the tested carbon filament yarns at higher temperatures starting from 500°C oxidation effects can occur, which might lead to an early failure.

The tensile tests were carried out in accordance with DIN 2062, with a slightly larger clamping length of 550 mm due to the constructional design of the heat chamber. In Table 3, the machine-related parameters are listed.

The results of the tensile force analysis on the AR-glass filament yarns are presented in Figure 5. On the one hand, the influence of the coating leading to an increased strength can be noticed, while on the other,

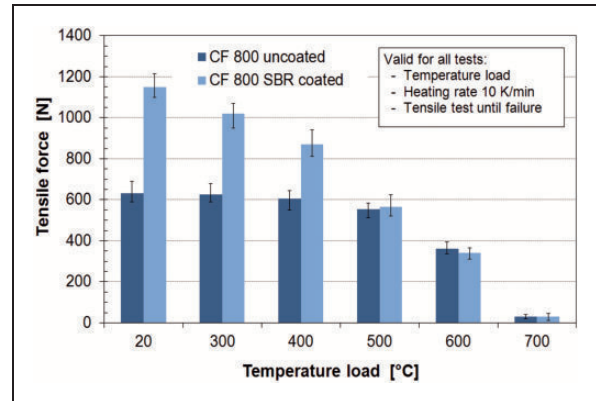**Table 3.** Machine and test parameters of the tensile test machine Zwick Z 100

Specification/technical data	
Clamping unit	Rope clamps
Length of the specimen	550 mm
Initial tension	0.5 ± 0.1 cN/tex
Test speed	200 mm/min
Heating rate	10 K/min

Figure 6. Tensile forces of carbon filament yarn 800 tex in dependence on the temperature.

Figure 5. Tensile forces of alkali resistant glass filament yarn 640 tex in dependence on the temperature.

it can be seen that there is a significant decrease in the tensile force under thermal loads. With an increasing temperature, the glass structure is slowly melted, due to the increasing movement in the inner molecular structure and therefore resulting in a decreased interactional force between the adjacent components. The consequence of this is a clear decrease in the tensile strength and elasticity module.

The melting of the glass structure starts at a temperature area between 400 and 500°C, as indicated by a significant decrease in the tensile force for the uncoated yarn at these test temperatures. For the coated yarns, the strength loss starts a lot earlier in comparison to the initial values under standard temperatures, which can be traced back to the melting of the polymer coating starting at about 200°C. In the continuing temperature curve, comparable strength values for coated and uncoated yarn after a completed decay of the coating could be recorded. A negative influence of the coating on the high-temperature behavior of the AR-glass filament yarn cannot be noticed.

The tensile behavior of the tested carbon filament yarn is presented in Figure 6. The coated yarns show almost double the strength of the uncoated ones under standard temperature due to the improved inner filament composite. Under thermal loads and exposure to the standard room atmosphere, oxidation effects occur

as expected and, therefore, the carbon is partly destroyed, which results in decreasing tensile strengths and stiffness.

The oxidation of the test specimen, which normally starts at temperatures of 500–600°C can be well proven by the resulting odor of the combustion gas (smell) and can also be backed up by the mass loss. Here, too, analogous with the AR-glass tests, the coated test specimens exhibit already a considerable strength loss at temperatures of 200–300°C, which results from the gradual melting of the coating. In the continuing temperature profile and after a complete decay of the coating, comparable strength values occur for uncoated and coated yarns starting at a temperature of about 700°C; the material is almost entirely destroyed. A negative influence of the coating on the oxidation and high-temperature behavior of the carbon filament yarn is not noticeable here either.

The tests also show the positive influence of the coating on the strength characteristics under standard temperature. As seen from the Figures 5 and 6, for both tested yarn specimens the maximum tensile force that the coated yarn can withstand is almost double that of the uncoated yarns. This can be attributed to the improved inner compound bonding between the single filaments. Because of the coating, the inner frictional interface between the adjacent filaments is increased whereby the core filaments located inside the cross-section inside are activated during the case of abrasive load (see Figure 7). A significantly improved utilization of the strength potential of the fibers is enabled by an even load distribution across the roving and significantly higher tensile forces can be transmitted.

Temperature-dependent bearing capacity under existing load

Under the predominantly prevailing working loads, which means under self-weight and the existing service
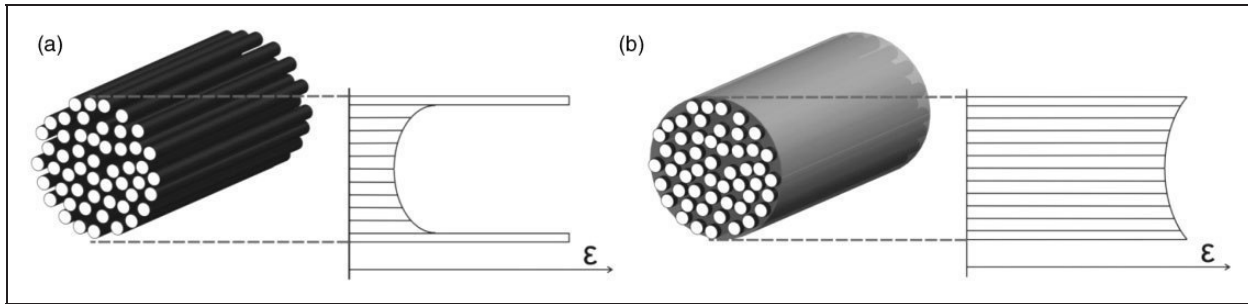
Figure 7. Coating-dependent tension distribution in the filament yarn according to Wiland et al.[29] (a) Filament yarn without impregnation. (b) Filament yarn with whole coating impregnation.

Table 4. Test plan for the temperature-dependent load capacity under existing load for sample carbon filament yarns
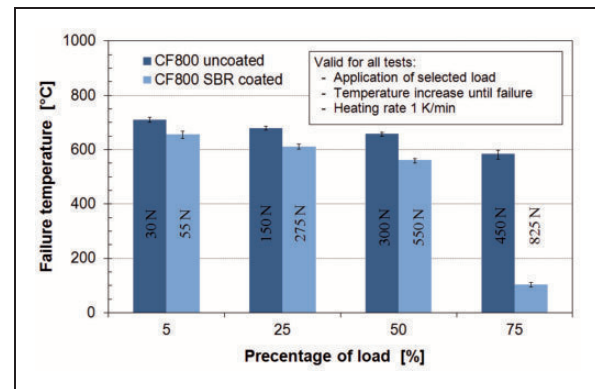
Loading percentage	CF 800 tex - uncoated	CF 800 tex - SBR coated
5%	30 N	55 N
25%	150 N	275 N
50%	300 N	550 N
75%	450 N	825 N
100% = max. tensile force	ca. 600 N	ca. 1100 N

SBR: styrol butadiene.

Figure 8. Temperature resistance of carbon filament yarns of 800 tex in accordance with the load level and in relation to the reference values under standard temperature.

load, tensions up to maximum 50% of the corresponding tensile strengths of the textile reinforcement (2.0-fold safety) are created for textile concrete components. In textile reinforcing layers, often the utilization of this strength potential is clearly lower. In extraordinary situations higher loads can, however, occur when several unfortunate impacts coincide under extreme conditions, such as in case of a fire.

In such scenarios, extremely high demands are made on textile reinforcements. It can be expected that the loading degree has a great impact on the temperature consistency of the corresponding yarn and therefore on the resistance durability of the entire construction. For the development of suitable safety concepts and the classification of the components according to different fire classification, detailed information about the behavior of textile reinforcements under different load levels at simultaneous high-temperature loads are essential.

The tests on the temperature-dependent load capacities under existing loads were carried out on carbon filament yarns of 800 tex uncoated and coated with SBS. The filament yarns were tested with a gradually increasing load level of 5% to 75%. The reference values of the subsequent tensile forces recorded under a standard temperature of 20°C (according to a 100% load level) for the uncoated and coated yarns at

different load levels derived from that for the tests are summarized in Table 4.

The aim of the test was to observe how the loaded yarns can resist the heat impacts under room air conditions, in case of fire. For this, the test specimen was first loaded with a tensile force and then heated at a rate of 10 K/min until failure. The duration until the break and the failure temperatures observed during the process provide insights on the temperature-dependent load capacities for different degrees of the impacting mechanical loads and the corresponding fire classifications.

Since the reinforcing textiles in their practical application as load bearing components are always under tensile stress due to their self-weight, the load level plays a significant role for the carrying behavior of such yarns under temperature loads. The influence of the mechanical load on the thermal consistency is presented in Figure 8, and it can be noticed that the failure temperatures tends to decrease with an increasing load level.

This behavior is distinctively higher for the coated yarns than for the uncoated ones, which can be derived

back to the melting of the coating and therefore limiting the compound behavior inside the yarn. Since the corresponding load level relates to the failure loads of the uncoated and the coated yarns, an early failure can be noticed at a higher degree of loading, as in the case with 75% of the failure load on the coated yarns. This is due to the fact that with increasing temperature, the coating melts and thereby loses its functionality. This results in overloading and rapid failure.

Temperature-dependent residual strength of pre-loaded yarns

Besides the knowledge about the temperature-dependent load capacities under existing loads, knowledge about the residual strengths at various temperatures on a pre-loaded yarn as well as under impact periods of different lengths are of great interest. This information allows statements about the specific loading capacity of the textile and therefore the component's behavior during the course of the fire.

The tests are carried out on uncoated and coated carbon filament yarns of 800 tex with the same load level according to Table 4, whereby the test specimen is heated until the desired test temperature is reached with a heating rate of 10 K/min after the application of the tensile force. Afterwards, the test temperature is kept constant during the defined impact duration and the load is increased subsequently until failure. The analyzed test scenarios are summarized in Table 5. Five single tests were carried out for each combination.

As stated before, the oxidation reaction taking place inside the carbon is mainly dependent on the influential parameters, such as the surrounding temperature and duration of the thermal impacts. The higher the temperature, the more kinetic energy is generated in the carbon molecules and more oxygen molecules can take part in the oxidation reaction. Hence, the reaction curve is accelerated, leading to a faster mass reduction and therefore to an early failure.

Besides the temperature, the impact duration also has an influence on the amount of oxidation reaction. The longer carbon is subjected to a corresponding temperature impact, the longer the oxidation reaction takes place and hence more material is destroyed. Hence, at temperatures of 300–400°C, during which the uncoated carbon filament yarns according to Figure 6 do not show any significant strength changes, they exhibit a decrease in strength when exposed to longer time periods under load. At temperatures of about 500°C and subsequent impact duration of 30 minutes, a strength loss of about 25% can be noticed for uncoated and coated yarns. At temperatures of 600°C and 700°C, the strength decreases entirely to zero after a certain period of time, which means under these temperatures,

Table 5. Test plan for the determination of temperature-dependent remaining strengths

Loading percentage	Test temperature	Time of exposure
0%	300°C	1 min
5%	400°C	5 min
25%	500°C	15 min
50%	600°C	30 min
75%	700°C	

the carbon filament yarns are completely destroyed due to the long impact durations (see Figure 9).

Tensile strength under thermal loads in an inert atmosphere (nitrogen)

These tests were carried out under an inert gas atmosphere to study the influence of the atmospheric conditions on the oxidation behavior of the carbon filament yarn and thereby to determine the temperature-dependent residual strength of uncoated carbon filament yarns. For this purpose, the yarns were heated analogous to the method described in the previous section with a heating rate of 10 K/min up to the test temperature. This temperature is then maintained for a defined exposure time and only then the actual tensile test was carried out. The test specifications are listed in the Table 6.

When compared to the test carried out under room atmosphere it can be observed here that the temperature level and the load duration aside, the oxygen content available is essential for the oxidation behavior and the related decrease in strength. The higher the existing oxygen amount, the more intensive is the oxidation reaction.

However, if the matrix or atmosphere surrounding the carbon roving does not contain any oxygen, no oxidation reactions take place under those inert conditions and the material exhibits unchanged structure and tensile (strength) characteristics even with temperatures above 1000°C. This theory could be validated with our tests carried out under nitrogen atmosphere. Tensile forces of the carbon filament yarns are compared with the experimental results from the developed test methods under both the room and inert nitrogen atmospheric condition, shown in Figure 10.

It can be clearly seen here that beside the resulting high temperature, the exposure time too has a significant influence on the significance of the oxidation reaction. The longer the carbon is exposed to the temperature impact, the longer the oxidation reaction takes place and more material is destroyed. Hence, already at temperatures in the range 300–400°C,
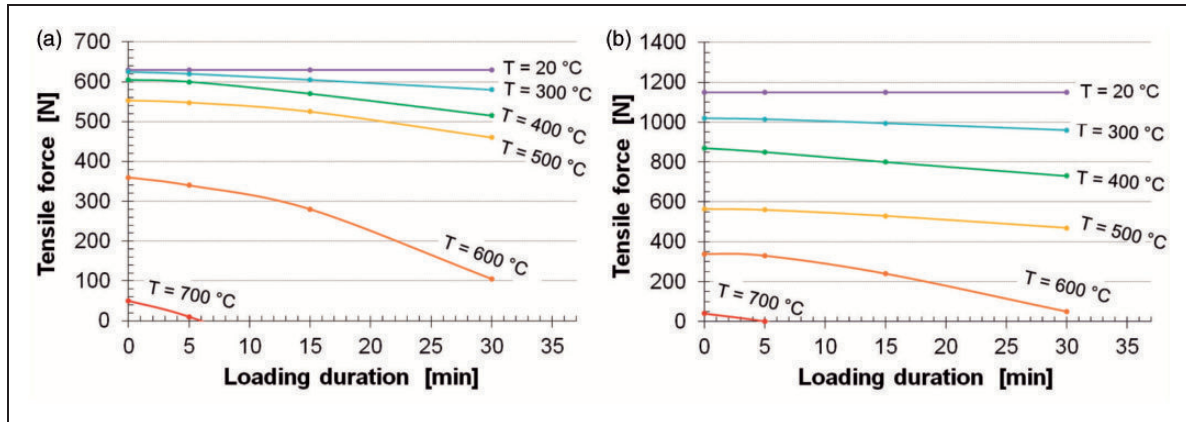
Figure 9. Tensile forces of carbon filament yarns of 800 tex in accordance with the temperature loads and impact duration: (a) behavior of uncoated yarns; (b) behavior of yarns with styrol butadiene coating.

**Table 6.** Test plan of the tensile tests on carbon filament yarns under a nitrogen atmosphere

Material	Atmosphere	Heating rate	Test temperature	Exposure time
CF 800 tex	– Atmospheric oxygen	10 K/min	300°C … 700°C	30 min

strength losses can be first noticed, which are in contrast to the results in Figure 6. From the temperatures above 500°C, an exposure time of 30 minutes results in a loss of 25% strength of the test specimen. At temperatures around 600°C and 700°C, the strength still decreases and after a certain time it reaches completely to zero, which means that under these temperatures, the carbon filament yarns are completely destroyed after a long exposure time.

However, under a nitrogen atmosphere, the tested carbon filament yarns themselves show no strength loss under the maximum possible test temperature of 700°C. The temperature loads are kept constant for more than 30 minutes without any decrease in strength or deformations of the test specimen.

Thermo-gravimetric analysis for the mass loss of carbon filament yarns

The thermo-gravimetric analysis (TGA), also called thermo-gravimetry, is an analytic method during which the mass changes of a medium to be tested are determined as a function of the temperature and the time under a defined atmosphere. The temperature increase occurs in defined time gradients. With this, the oxidation behavior of the yarn and the decay behavior of the coating under different heating scenarios and atmospheric conditions are determined for the case of carbon filament yarns. In this case, the test specimen is heated to the desired test temperature in a little aluminum oxide skillet with a defined heating
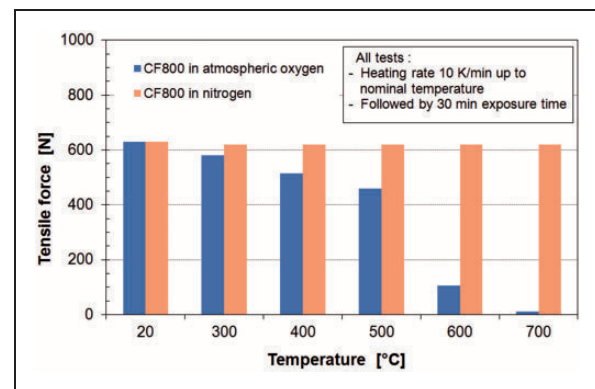
Figure 10. Temperature-related tensile forces of carbon filament yarns of 800 tex under oxygen-containing room and nitrogen-containing inert gas atmosphere.

rate, whereby the heating chamber is hermetically sealed and can optionally be flooded with nitrogen. The test specimen container (skillet) is connected to a micro-scale, which records the mass changes during the heating process. The TGAs took place at the Leibniz-Institute for polymer research Dresden e. V. (IPF) in accordance with DIN 51006. However, these tests were carried out divergent to the recommended test amount of 10 mg with a net weight of 6 mg, due to limitations in the dimension of the skillet.

Uncoated and coated carbon filament yarns were analyzed under different test temperatures and different heating rates under oxygen-abundant room air

Table 7. Overview of the test material and boundary conditions of the thermo-gravimetric analysis (TGA) experiments

Test specimens and boundary condition for the TGA tests	
Material	CF 800 tex – uncoated CF 800 tex – SBR coated
Atmospheric condition	– Atmospheric oxygen – Nitrogen
Measurement	Continual measurement from the norm temperature until the target temperature

SBR: styrol butadiene.

Table 8. Thermo-gravimetric analysis (TGA) tests plan on the mass loss of carbon filament yarn under different temperatures under room conditions

TGA tests under room conditions/Varying target temperatures					
Heating rate	500 K/min				
Test duration	180 min (after the target temperature)				
Target temperature	300°C	400°C	500°C	600°C	700°C

Table 9. Thermo-gravimetric analysis (TGA) tests plan about the influence of the heating rate on the mass loss of carbon filament yarn under room conditions

TGA tests under room conditions/Different heating rates						
Heating rate	2 K/min	5 K/min	10 K/min	20 K/min	40 K/min	80 K/min
Test duration	340 min	136 min	68 min	34 min	17 min	8.5 min
Target temperature	700°C					

Table 10. Thermo-gravimetric analysis (TGA) tests plan about the mass loss of carbon filament yarn under nitrogen

TGA tests under nitrogen	
Heating rate	10 K/min
Test duration	180 min (after the target temperature)
Target temperature	1000°C

temperature as well as under nitrogen, whereby in most cases a continuing mass determination was carried out. The single test series with a variation of different test parameters and marginal conditions are presented in Tables 7–10.

In order to verify the observed temperature-dependent characteristics in the tensile tests, the subsequent results determined using TGAs were compared initially for the standard room atmosphere conditions. Initial reductions of about 1% in mass could be noticed in a temperature area starting at about 250–300°C for the uncoated yarns and about 8–10% for the yarn coated with SBS, which summarizes the decay of size and coating (see Figure 11), whose mass shares are almost exactly in accordance to these values.

At about 400–450°C, the decay of the mass and coating is complete. In a continuing temperature curve, the mass reduction continues rapidly depending on the heating rate under room air temperature. At about 500–600°C the material is completely destroyed. For a slow temperature increase and a longer impact duration, the mass reduction starts earlier, while for a rapid temperature increase and a shorter impact duration the mass reduction is delayed.

Furthermore, it could be noticed that for coated yarns after the decay of the coating, the continuing oxidation-based mass loss of the carbon already takes place slightly earlier, which points to an accelerating effect of the coating on the oxidation process. Therefore, the noticed mass losses correlate to their noticed strength losses under the corresponding temperatures after the tensile force tests.

Similarly, the TGAs with uncoated and coated carbon filament yarn under a nitrogen atmosphere were carried out with a test temperature of up to 1000°C (see Figure 12). With respect to the decay of the size and coating, the same effects can be noticed
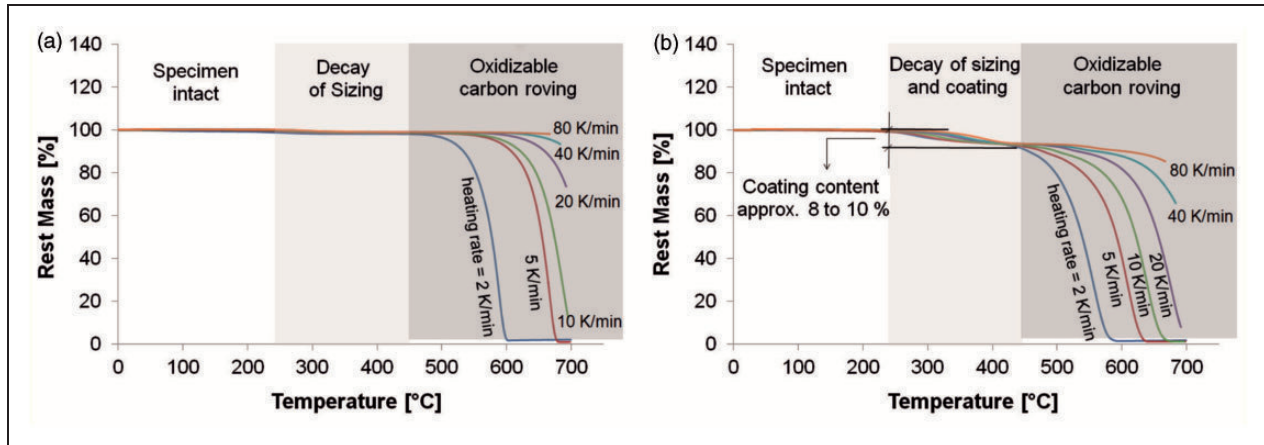
Figure 11. Temperature-dependent mass reduction of carbon filament yarn under room air atmosphere for different heating rates: (a) mass reduction of uncoated yarn; (b) mass reduction of yarn coated with styrol butadiene.
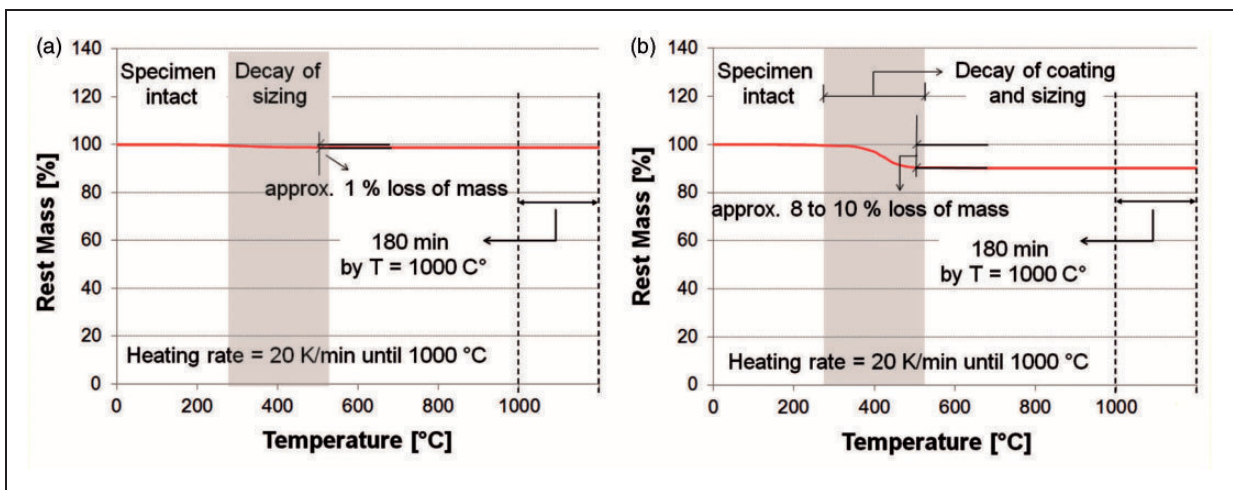
Figure 12. Mass reduction of carbon filament yarns under nitrogen with a test duration of three hours under a temperature of 1000°C: (a) mass loss of uncoated yarn; (b) mass loss of yarns with styrol butadiene coating.

here, which means a mass reduction of about 1% and 8–10% takes place at temperatures of about 250 and 300°C, respectively. With a continuing increase in temperature, a constantly consistent mass reduction was noticed in all cases, which means no further reactions could be recorded up to a maximum test temperature of 1000°C. These results, too, correlate exactly with the tensile force tests.

In addition to the TGAs, further analyses were carried out to measure the mass reduction after an initial sudden increase in temperature (500 K/min) and then subsequently maintaining a constant temperature (see Figure 13). In the case of coated test specimens, it could be seen that already at 400°C, a slight mass reduction took place, while at 500°C and after impact duration of 30 minutes, there was already a mass reduction of 25%. The test specimens at temperatures of 600°C as well as

700°C were already completely destroyed after a short amount of time, which means there was no remaining mass. These measurements correlate with and prove the temperature-dependent strength losses noticed in the tensile force tests.

Summary and prospects

The work described in this paper forms the basis for the determination of tensile mechanical behavior and the creation of a meaningful characteristic database for the characterization of the textile high-performance fiber material under thermal loads.

As a first step, it was made possible to develop a novel and reliable measuring method with a reproducible tensile force tests using high-temperature impact loads. Using an infrared radiator and the suitable
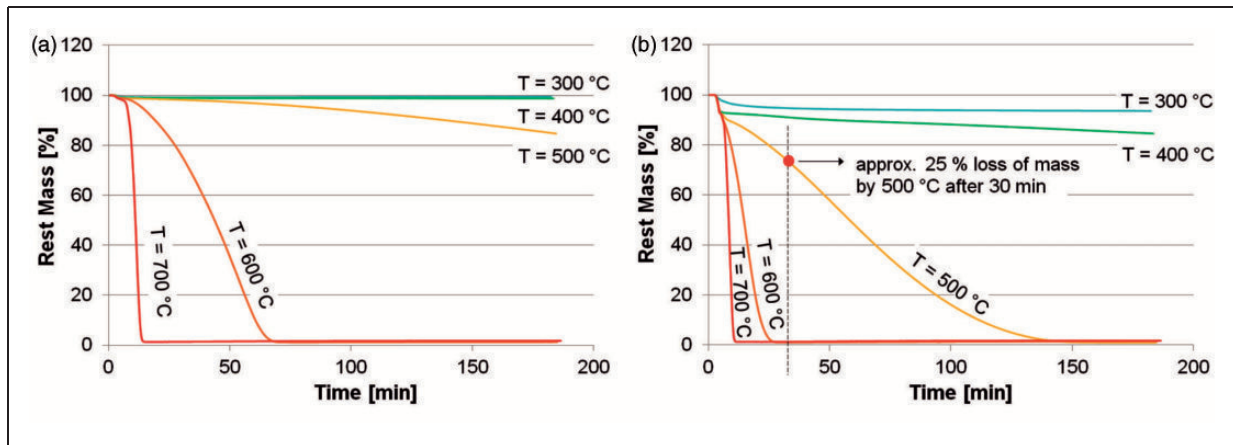
Figure 13. Time-dependent mass reduction of carbon filament yarn under room air atmosphere at different test parameters: (a) mass loss of uncoated yarn; (b) mass loss of yarn with styrol butadiene coating.

control, filament yarns can be tested under temperatures of up to 700°C. Furthermore, the developed test stand with optional inert gas connecting tubes allows the carrying out of tensile tests under inert atmospheres, that is, under protection gases such as nitrogen.

With the help of the performed tests on AR-glass and carbon filament yarn, it was made possible to extensively characterize their strength characteristics during impacting thermal loads under different conditions and test scenarios. A reliable characteristic value database on the material behavior of these high-performance fiber materials under high-temperature loads can thus now be established. With the experimentally determined values, significant influencing parameters on the materials, such as the impacting temperature, the heating rate and the load degree, could be determined and classified.

It was noticed that for carbon filament yarns, the surrounding medium and its chemical composition played a significant role with respect to their thermal consistency. At temperatures of 300–400°C under the influence of oxygen, oxidation reactions occurred resulting in a gradual material damage and thereby decreasing strength. In contrast, carbon filament yarns show an extremely high thermal stability under inert conditions at temperatures far more than 1000°C, as could be shown with several tests under nitrogen atmosphere.

Moreover, the fiber material was not only analyzed with respect to the uncoated condition as delivered by the producer, but numerous tests were also carried out with coated yarns to specify the influence of polymer coating applications on the strength characteristics under thermal loads. These coatings play an important role in the improvement of the tensile mechanical characteristics, hardening and structure stabilization of textile semi-finished products. Thus gaining information about their thermal potential and their behavior

under thermal loads is highly relevant. Thus, not only the thermal characteristic of existing coating agents can be determined and validated but also suitable requirement guidelines for the development of futuristic and thermally consistent coating formulations can be precisely defined.

Funding

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