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Effects of temperature and plasma treatment on mechanical properties of ceramic fibres

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Properties

ABSTRACT

Purpose: The aim of this study is an investigation and comparison of mechanical properties of ceramic fibres after they were influenced by temperature and plasma treatment.

Design/methodology/approach: Single filament after being processed at different temperatures (200°C, 400°C, 700°C and 1000°C) and methane plasma treatment was separated with a magnifier, prepared on a punched mounting tab, and was evaluated in accordance with Japanese Industrial Standard.

Findings: Preliminary results of the improvement in tensile strength, Young's modulus, elongation of ceramic fibres after plasma treatment are studied in this paper.

Research limitations/implications: The samples were tested for optimized parameters of plasma modification and optimized parameters of plasma to ceramic fibres curing. There was not enough time to test the adhesion between Geopolymer matrices and ceramic fibres.

Practical implications: In the future, our work will be focused on optimization of parameters for plasma modification of fibres made of different materials and applying this method to improve the fibre and Geopolymer matrix adhesion.

Originality/value: The value of this work is defined by the influence of plasma treatment parameters on quality, mechanical properties of ceramic fibres and increasing the adhesion between matrices and reinforcements.

Keywords: Materials; Composites; Engineering Polymers; Biomaterials; Technological Devices and Equipment; Aramid; Silicone; Application

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1. Introduction

Ceramic fibres are typically made from alumina, silica, and other metal oxides or, less commonly, of non-oxide materials such as silicon carbide. Most ceramic fibres are composed of alumina and silica in an approximate 50/50 mixture [3]. The main subject of this paper was focused on the 3M Nextel 312 fibre 400tex, which first appeared in 1974, it is composed of 62% wt Al_2O_3, 24% SiO_2 and 14% B_2O_3. It has an essentially amorphous structure and it is limited to be used below 1000°C because of the volatility of boria but it remains in the foundation of the 3M Nextel range of oxide fibres [5]. Oxide fibres, currently available commercially, are mostly based on Al_2O_3 - or Al_2O_3/SiO_2 ceramics. They possess high values for tensile strength and modulus, and due to their oxidic nature they are stable against oxidation at high temperatures [3]. Table 1 shows a detail of the typical properties of alumina silica based fibre 3M Nextel 312.

Disadvantages of ceramic fibres are their susceptibility to oxidation, which leads to fibre degradation in oxidizing atmosphere over time, so we used the plasma method to improve some fibres properties i.e. debility, oxidation and mechanical properties.

Features of 3M™ Nextel™ fibres:

- Low elongation at operating temperatures
- Low shrinkage at operating temperatures
  Good chemical resistance
- Low thermal conductivity
- Thermal shock resistance
- Low porosity
- Unique electrical properties [5].

2. Experimental

2.1. Mechanical properties of ceramic filaments at different temperatures

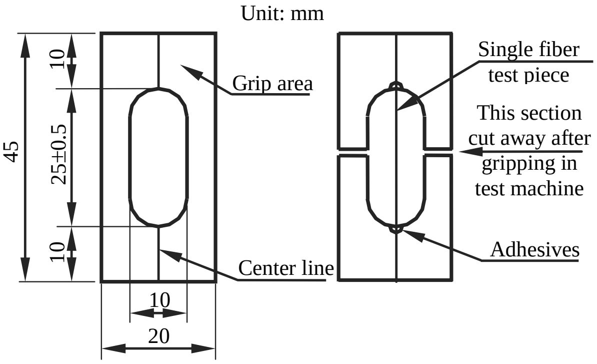

Fig. 1. Mounting tab for single filament testing

Ceramic fibres were put in the furnace at 200°C, 400°C, 700°C and 1000°C for 3 hours with a gradient of 10°C/min. The rate of temperature velocity was 283.15 K per minute (873.15 K/hour), and then fibres were cooled in the furnace by opening the gate [1].

Single filament of each ceramic fibre were separated with a magnifier and prepared on a punched mounting tab. The single filament test piece was bonded by an adhesive so as to let the length specified gauge under the condition make the filament straight along the centre line of the mounting tab. This was evaluated in accordance with Japanese Industrial Standard (JIS R 7601) [2]. Tensile strength and Young's modulus were calculated from the load-elongation records and the cross-sectional area measurements. The specimen is shown in Fig. 1.

The samples were tested by the Instron LaborTech 2.050 machine (maximum sensor load: 5 N).

Table 1.
Properties and composition of alumina silica based fibre 3M Nextel 312

Composition [wt%]	Diameter [µm]	Density [g/cm^3]	Strength [GPa]	Strain [%]	Young's Modulus [GPa]	CTE* [10^{-6}/C]
62% Al_2O_3 24% SiO_2 14% B_2O_3	10÷12	2.7	1.7	1.12	152	3(25 -500°C)

*CTE: Coefficient of thermal expansion

2.2. Mechanical properties of ceramic filaments after plasma treatment and continuous curing at different temperature

Radio frequency (RF) plasma reactor was employed for the plasma surface treatment of ceramic fibres. The RF energy is supplied to the system through an isolated electrode, which is powered by a 13.56 MHz (1200 W maximum power) home-made matching network. The reaction chamber is made of stainless steel tubing 1200 mm high and 280 mm inner diameter. The system is additionally equipped with a flow controller at gas inlet, a pressure gauge and rotary vacuum pump.

The process of plasma treatment was described as follows: The fibres were placed on a substrate holder inside the deposition chamber and were exposed to methane low-pressure glow discharge plasma in the 1 - 20 min range. Methane flow rate from 10 sccm to 30 sccm was used and the working pressure inside the plasma chamber was maintained in the 25 - 40 Pa range. Negative bias voltage (V_b) varied from 100 to 900 V.

Ceramic fibres were put in the furnace at 200°C, 400°C, 700°C and 1000°C for 3 hours with a gradient of 10°C/min, and the process was repeated using the same method with section 2.1. Fig. 2 shows the process for treatment of fibres.

3. Results and discussion

3.1. Mechanical properties of ceramic filaments after heat treatment at different temperatures

Table 2 shows values for tensile strength, Young's modulus and elongation of ceramic fibre. The tensile strength at 20°C had a higher value than the producer from 80% to 85%, who reduced Young's modulus value from 152 MPa to 140 MPa (Table 1).

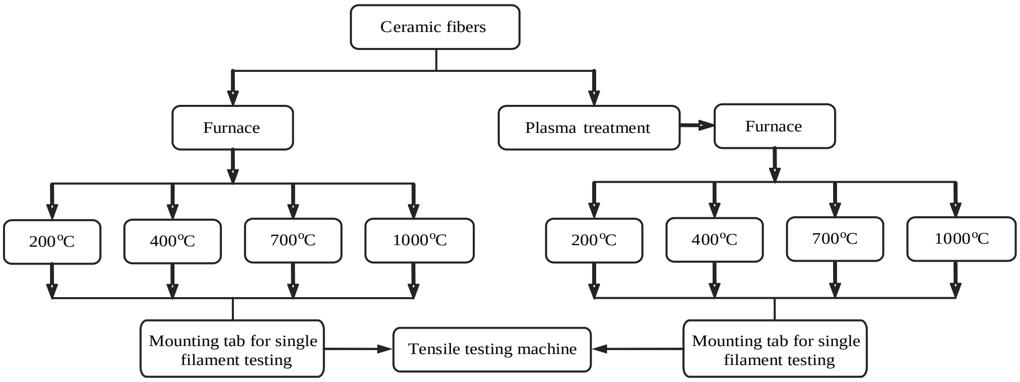

Fig. 2. The process for treatment of fibres

Table 2.
Mechanical properties of ceramic filaments under different temperature

20°C			200°C			400°C			700°C			1000°C		
A [%]	R_t [MPa]	E [GPa]	A [%]	R_t [MPa]	E [GPa]	A [%]	R_t [MPa]	E [GPa]	A [%]	R_t [MPa]	E [GPa]	A [%]	R_t [MPa]	E [GPa]
1.48	1995	140	2.06	2378	128	1.36	1745	117	1.39	1818	123	0.82	1161	99.7

Table 3.
Mechanical properties of some kind filaments under different temperature [1]

Kind of fibre	Average diameter [μm]	20°C			200°C			400°C			700°C		
		A [%]	R_t [MPa]	E [GPa]	A [%]	R_t [MPa]	E [GPa]	A [%]	R_t [MPa]	E [GPa]	A [%]	R_t [MPa]	E [GPa]
Carbon HTS 5631 800tex 12K	7	1.75	3120	178	1.72	3120	181	2.24	3640	163	Two kinds of the fibres were destroyed totally (nearly disappeared)		
Carbon HTS 5631 1600tex 24K	7	1.84	3120	170	1.33	2340	176	1.66	2861	172			
AR 2400tex	27	3.32	1293	39	3.22	1241	39	1.63	769	47	The fibres still remained in the furnace, but so brittle		
ARG 2500tex	14	2.68	1560	58	2.24	1820	81	0.63	390	62			
Basalt BCF13 - 2520tex - KV12 Int	13	3.98	2563	64	3.44	2111	61	1.7	1281	75			
E-glass 2400tex	24	4.72	1504	32	3.26	1106	34	2.08	995	48	1.03	575	56

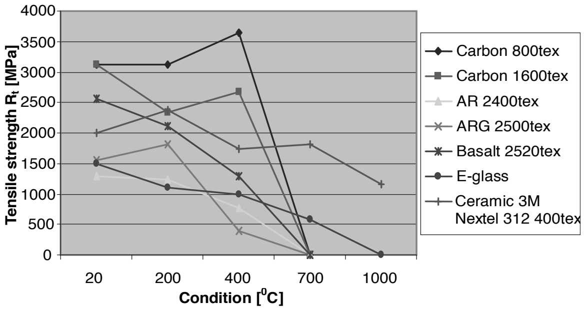

Fig. 3. Effect of temperature on tensile strength of some kinds fibres

The authors think the producer tested under standard conditions and using a different velocity, while the authors used a velocity v = 5 m/s to test ceramic filaments. The tensile strength of ceramic fibres at 200°C had higher values than other temperatures, while continuous increasing thermal processing reduced values of Young's modulus.

Comparison of mechanical properties of ceramic fibres with some different kinds of fibres is presented and described in table 3. After 3 hours sustained at 200°C the tensile strength, elongation and Young's modulus of all kinds of fibres are approximately the same as before. The only exception being E-glass fibre, which presented nearly 73% strength (1106 MPa compare to 1504 MPa) and elongation of this fibre reduced from 4.72% to 3.26% comparing to that of at room condition.

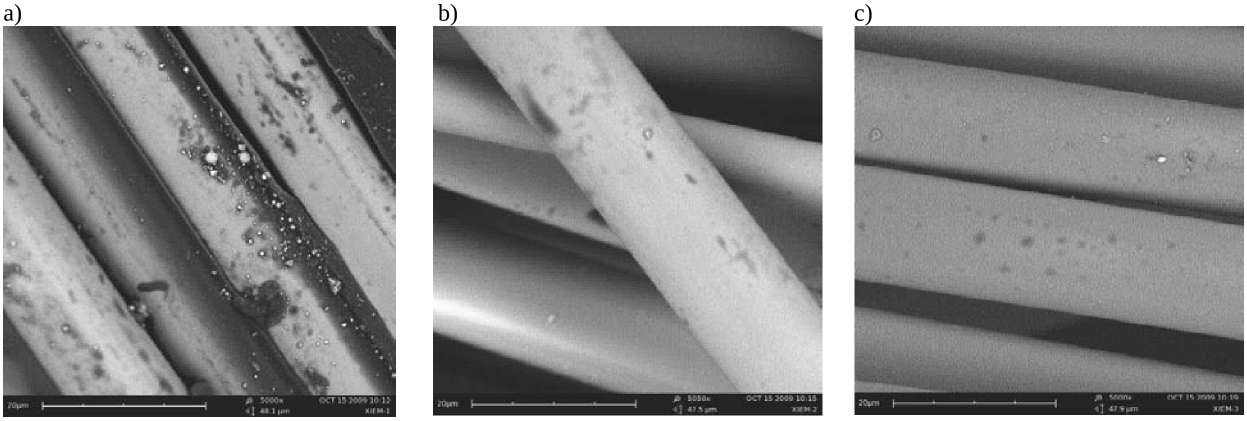

Fig. 4. Scanned electron microscope images of the surface of ceramic fibers under heat treatment: a) 20^{o}C, b) 400^{o}C, c) 1000^{o}C

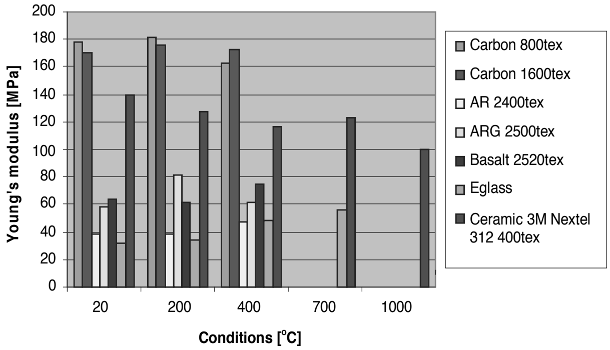

Fig. 5. Effect of temperature on Young's modulus of some kinds fibres

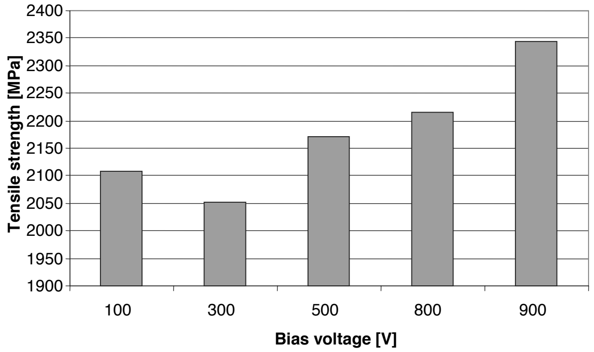

Fig. 6. Effect of bias voltage on tensile strength

It can be seen from Fig. 3 and Fig. 5 that the tensile strength value of all fibres drop sharply after 400^{o}C and until 700^{o}C most of the fibres were destroyed completely or still remained in the furnace, but were very brittle, while ceramic fibres were decreased slowly. Results of the investigation indicate that ultimate tensile strength decreases with temperature increase. In all cases, the Young's modulus (Fig. 5) and elongation decreases as temperature increases. Ceramic fibres are preferred because of the stability of the strength in high temperature.

Fig. 4 displays SEM images of the original fibers and fibers after heat treatment at 400^{o}C and 1000^{o}C for 3 hours. If the heat-treatment temperature increases to 1000°C, these surfaces of ceramic fibres were increased smooth.

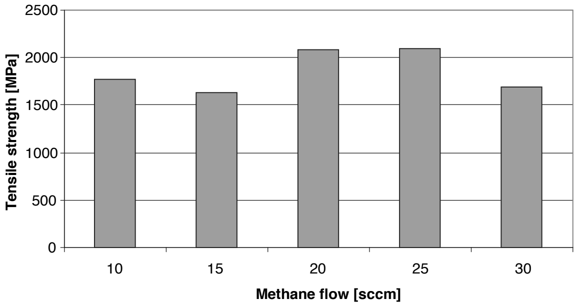

Fig. 7. Effect of methane flow on tensile strength

3.2. Optimization parameters of plasma

There were three main plasma treatment parameter effects to mechanical properties on the surface chemical composition of the ceramic fibres; namely the treatment time, flow rate of methane and bias voltage. The results are shown in Fig. 6 and Fig 7.

It can be observed that high bias voltage can increase the tensile strength and the contrast, increasing time of plasma treatment, would cause a decrease in tensile strength.

In bias voltage, while keeping the RF = 1000 W, pressure p = 25 Pa, treatment time (t = 7 min) and methane flow rate (CH_4 = 20 sccm), the values of tensile strength were highest in V_b = 900 V (Fig. 6). This indicates an optimal bias voltage of about 900 V. When the treatment time, RF power, pressure and bias voltage are kept constant while increasing the methane flow rate, the authors easily determined the methane flow rate was optimized at around 25 sccm (Fig. 7). Thus optimization parameters of plasma to curing ceramic fibres were p = 25 Pa, t = 7 min, V_b = 900 V and CH_4 = 25 sccm.

Table 4.
Mechanical properties of ceramic filaments after plasma treatment and continuous heat treatment at different temperature

20°C			200°C			400°C			700°C			1000°C		
A [%]	R_t [MPa]	E [GPa]	A [%]	R_t [MPa]	E [GPa]	A [%]	R_t [MPa]	E [GPa]	A [%]	R_t [MPa]	E [GPa]	A [%]	R_t [MPa]	E [GPa]
1.52	2092	141	1.88	2386	149	1.39	2065	138	1.24	1990	145	0.81	1631	189

3.3. Mechanical properties of ceramic filaments after plasma treatment and continuous heat treatment at different temperatures

The treatment parameters were studied to find the best suitable condition. The testing was performed on tensile strength, Young's modulus and elongation until the break of the fibre.

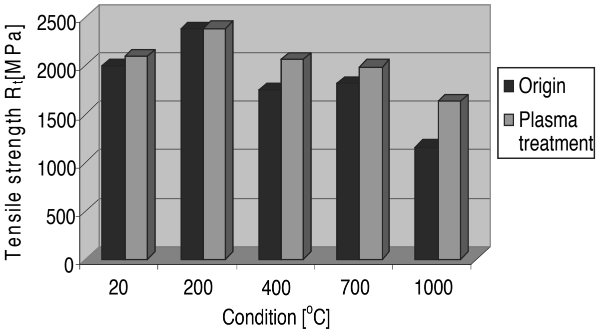

Fig. 8. Effect of temperature and plasma treatment on tensile strength of ceramic fibres

After methane plasma treatment, the tensile strength and Young's modulus of ceramic fibres were increased to greater than that of the original fibres. The tensile strength at 400°C had a higher value than the heat treating fibres by about 84%; however elongation at 200°C and 700°C were decreased slightly (Table 4).

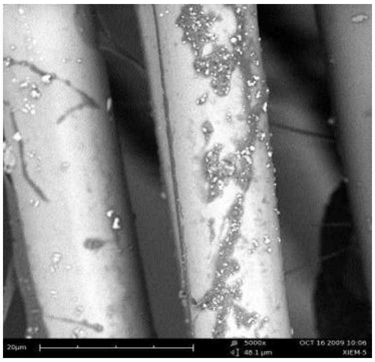

Fig. 9. SEM images surface of ceramic fibres after plasma treatment

Compared to treatment by silane coupling agent, methane plasma treatment can improve both the mechanical properties of fibre and adhesion, while silane coupling agent only improves adhesion.

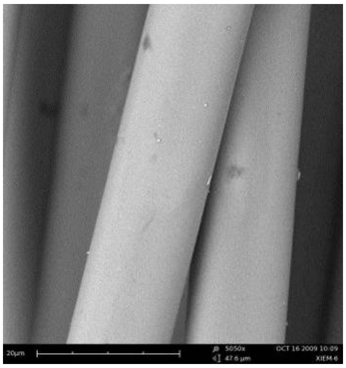

Fig. 10. SEM images surface of ceramic fibres after plasma treatment and continuous heat treatment at 1000°C

Fig. 9 show that these surfaces of ceramic fibres were rougher surface than the original fibres. This is believed to be one of the main reasons to improve the fibre and matrix adhesion. Fig. 10 presents the surface image of ceramic fibres after plasma treatment and continuous heat treatment at 1000°C. This image delineated much smoother surfaces of fibres.

4. Summary

The conclusions of the present research can be stated as follows:

Heat-treatment had dramatic effect on the microstructure and the phase composition of the surface of ceramic fibres at different temperature. The surfaces of ceramic fibres were increased smoothly if the heat-treatment temperature increased below 1000°C. Surfaces of ceramic fibres at 200°C can improve the mechanical properties of composites, both flexural strength and elastic modulus; heat treatment at 1000°C causes little improvement for the mechanical properties.

The authors tested samples to optimal parameters of plasma to cure fibres. Composites prepared with surface plasma treated fibres have shown generally much better wetting and adhesion than those made from untreated fibres and improved mechanical properties of the composites. In the future, our work will apply this method to improve on the fibre and Geopolymer matrix adhesion.

Acknowledgements

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References

[1] T.D. Hung, D. Kroisová, O. Bortnovsky, P. Louda, N.T. Xiem, Preliminary abilities of thermal sustainment of composites based on geopolymer matrices, Book of Abstracts of 3rd International Conference on Vacuum and Plasma Surface Engineering. Liberec, Technical University of Liberec, 2008.

[2] Translated and Published by Japanese Standards Association, JIS R 7601 - Japanese Industrial Standard - Testing Methods for Carbon Fibbers, Japan, 1986.

[3] Ceramic Fibbers (Respirable Size), Reasonably anticipated to be a human carcinogen, First Listed in the Seventh Annual Report on Carcinogens, 1994.

[4] Narottam P. Pansal, Handbook of ceramic composites, Kluwer academic publishers, USA, 2005.

[5] http://www.directindustry.com/prod/3m-manufacturing-and-industry-ceramics/ceramic-fiber-17950-39618.html, accessed: 10/9/2009.

[6] I. Niewiadomska, K. Przybył, Plasma method in textiles, Book of Abstracts of 15th International Conference, Structure and Structural Mechanics of Textiles, Organized by Faculty of Textile Engineering, Technical University of Liberec, Czech Republic.

[7] S.M. Rossnagle, J.J. Cuomo, W.D. Westwood, Handbook of plasma processing technology, 1990.

[8] Y. Seki, K. Sever, M. Sarikanat, H.A. Gülec, I.H. Tavman, The influence of oxygen plasma treatment of jute fibres on mechanical properties of jute fibre reinforced thermoplastic composites, Proceedings of the 5th International Advanced Technologies Symposium (IATS’09), 2009, Karabuk, Turkey.

[9] V. Chirila, G. Margineana, W. Brandl, Effect of the oxygen plasma treatment parameters on the carbon nanotubes surface properties, Surface & Coatings Technology 200 (2005) 548-551.