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Non-standard test methods for long-fibrous reinforced composite materials

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ABSTRACT

Purpose: The investigations serve as comparative verification of silicone-aramid material designed for oesophageal prosthesis.

Design/methodology/approach: In this work, non-standard research techniques, particularly designated for comparative tests of long-fibrous composite materials, are presented. Those tests are carried out on equipment similar to so called NOL rings test and pressure tests using rubber as a working medium

Findings: During interpretation of pressure tests results according to the proposed method, it should be paid attention to the state of stresses in the wall of investigated tubular element. Ignoring fractional resistance of the rubber with sample wall, it can be obtained uniaxial stress in circumferential direction.

Research limitations: Proposed research methods (non-standard NOL rings and tests using axial compressed rubber) can be applied in evaluation of fabricated long-fibrous material, e.g. determination of degree of silicone material binding.

Originality/value: Proposed methods of comparative tests are easy to make and the destruction point during static tests as well as fatigue ones is clearly determined. Results of investigations serve as comparative verification of silicon-aramid material designed for oesophageal prosthesis. Tested tubular element - prototype of oesophageal prosthesis requires the reduction of reinforcing material.

Keywords: Composites; Engineering polymers; Aramid fibres; Silicone, Biomaterials; NOL rings

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MATERIALS

1. Introduction

Composite materials are the most promising group of engineering materials and they have been the subject of many research programs. Furthermore, they are successfully applied in industry. Nowadays, requirements of technique, industry and medicine have proved that properties of basic groups of engineering material are limited and insufficient in many cases. Realization of technological goals which arises in front of modern engineers would be impossible without contemporary development of advanced materials, which are composite materials, often shortened to composites. Those materials are widely applied in medicine, where special emphasis is put on modern engineering materials with very specific properties that are achieved by combination of various components. Among described materials, fibrous composite materials reinforced with continuous filament are undoubtedly an interesting group [1-7,18-25].

For last four decades, there have been fundamental progress of properties of materials reinforced with continuous filament components which caused extension of their application field. Within the framework of development of proper systems of connection and production processes, cooperation of economic factors with maintenance of corrosion resistance and decrease of element weight is recommended. The following methods of manufacturing of tubular elements reinforced with continuous filament have been used up to now: coiling up, plaiting, winding and hand forming method, However, those methods have been of little importance in industrial practice. What is more, those methods are limited in different ways: long time of manufacturing, length and shapes, and some of them also high cost of production. For those reasons, proper making of reliable samples for preliminary tests is so important, especially in the case of newly worked out materials with the usage of expensive components. In particular, this guidelines refers to composite materials with specific medical application, where cost of single sample production is relatively high, and conditions of production are strictly determined by adequate procedures. It is a condition of necessity of making preliminary tests on a small number of samples, on the basis of which, it can be determined if the shape of given material is appropriate for its function. The crucial stage of designing techniques of prototype manufacture selection is verification of selected materials by tests, e. g.: strength ones. In this paper, There are presented two types of non-standard method of tests, that is with the usage of untypical NOL rings and pressure tests using rubber as a working medium. Both mentioned tests are usually used in Institute of Materials Science and Applied Mechanics, Wrocław University of Technology for evaluation of produced composite material [7,10-11].

Proposed shape of samples may be applied in torsion test, axial tension test, internal pressure test, etc., as well as their combinations.

2. Material

Wide range of polymers with medical application include such important components as silicones and continuous fibres. In XXI century plastics are everywhere. Most commonly, the continuously linked backbone of a polymer used for the preparation of plastics consists mainly of tetravalent carbon atoms. Nevertheless, silicones are different because they consists of silicon atoms. Those materials have been studied as a result of research of plastic satisfying far higher, specific requirements, not fulfilled by so far known materials of this type. Due to those researches, medical silicones have been developed, which are produced with proper care of high chemical purity in comparison to technical silicones. Applied procedures and purity requirements in production stage guarantee absence of substances, that could undergo biochemical reactions resulting in inflammatory conditions, irritations, allergy or cellular mutation while long-lasting contact. It is proven by the fact of application of silicones as implants and silicone scar sheets. Medical silicones are undoubtedly one of the few substances with so high biocompatibility what makes them even more attractive material. Unfortunately, these materials are not characterized by good mechanical properties. For that reason, their modification trials are very important. One of the experimental method of silicones reinforcement is introduction of reinforcing phase in the form of rigid and resistant continuous plastic fibres. In practice, there is important percentage of introduced fibres by volume, their adhesion to the matrix as well as biocompatibility of obtained composite material [3-5].

State of the art highly resistant plastic fibres are certainly aramid fibres. Aramid fibres are practically unresponsive to surface defects and resistant up to 300°C. Because of these reasons, the present application of aramid fibres in technology is very often and common, but there are rarely applied as the reinforcement in polymeric materials used in medicine. Despite their very good mechanical properties, low weight and low elongation there are not so readily used in medical industry as polyethylene, glass or carbon fibres, which are characterized by worse mechanical properties. However, most recently announced optimistic results of researches on biocompatibility of aramid fibres and their further prognoses positively influence on increase of interest of their implementation in widely understood medicine [2,5-7,11].

For the samples preparation following components have been used: aramid fibre - Kevlar 49 (DuPont USA) and medical silicone (DOW CORNING USA) (Tables 1,2). Components of composite material have been chosen taking relevant biochemical and mechanical properties of final product, like high biocompatibility, purity and adequate elasticity into consideration.

Table 1.
Properties of 2200 1610 aramid fibres

PROPERTIES OF ARAMID FIBRE					
Type of fibre	Density [g/cm^3]	Young modulus [GPa]	Tensile strength [MPa]	Ultimate elongation [%]	Breakdown temperature [°C]
2200	1.44	105	3053	2.70	490
Contraction in hot air (15 min at temperature 190°C) => 0.1 [%]					
Thermal resistance (48 h at temperature 200°C) => 90 [%]					

Table 2.
Chemical and physical properties of medical silicone manufactured by COW CORNING

CHEMICAL AND PHYSICAL PROPERTIES				
Boiling temperature/range [°C]	Flash-point [°C]	Specific gravity [g/cm^3]	Viscosity [cSt]	Physical state; colour; odour
> 82	13.3 (closed crucible Pensky-Martens)	0.865	132 (at temperature 25°C)	Liquid; straw-coloured; organic solvent

3. Methodology

Tensile static test consistent with quality standard ISO 6259/1-3 is commonly used method of mechanical properties evaluation of tubular elements. In this case, samples in the shape of so-called dumbbell are blanked from tubular element parallel to its axis. However, results obtained by this way do not reflect actual stresses in tubular element during work. Additionally process of preparing of a single sample is connected with occurrence of effects resulting from blanking process, that is surface roughness, dimensional tolerance or structural changes in blanking zone, etc.. Those tests are particularly inadequate in respect of samples made from long-fibrous composite material, where cross- linking and uncovering of fibres take place what causes notch effect inside investigated material. In long-fibrous composite materials it is very important factor that weakens the material, lowers the results and increases the scatter of their range. Besides there arises a problem of samples holding considering flat samples. For all mentioned reasons, authors have decided to used non-standard methodology of tubular samples and NOL rings researches [7-18].

Methodology of tubular samples preparation is classified as belonging to a category of relative simple methods in comparison with dumbbell preparation which is very difficult to make of long-fibrous composite materials with complicated system of fibres. Proposed samples are also characterized by better repeatability and accuracy of measurements [10-11,14].

Tubular samples used in described tests have been done on winding reel placed in Institute of Materials Science and Applied Mechanics, Wrocław University of Technology. As an reinforcement aramid fibres Kevlar 49 2200 1610 type manufactured by DuPont have been used. Reel of fibres have been placed in so-called rewinder with controlled tension. Tension approximately 5N has been used. Fibres have been saturated using specially constructed drum saturating system. It has been winded on with the fibre speed 0.2 m/s approximately, at an angle of ±45°.

Research method based on so-called NOL type rings is directed to more realistic mapping of the stresses reigned in investigated material during work. Fabrication (winding) and construction technology map the work of unidirectional composite material during elongation along the fibres Each sample is a separated steel disc (core and handle in the same time) with working diameter of 105 mm, on which strictly determined number of bundle of fibres saturated with impregnant are wound evenly using proper feed of winding reel support (Fig. 1a).In this way composite ring with diameter of 20 mm and required thickness is obtained. Next, ring-shape samples are hardened in hanging position loaded with weight of 1 kg et the ends of the bundles to provide uniform tension of fibre in the sample. On the Fig. 1B, there can be observed the sample which is articulately hang on testing machine MTS 810. Previously saturated aramid fibre have been wound on the sample 60 times (4 layers - 15 winds each) and it results in composites of 1.2 mm thickness and 20 mm in diameter.

4. Investigations

Following investigations have been done:

- tensile static test of NOL rings,
- pressure static test of tubular samples,
- pressure fatigue tests of tubular samples.

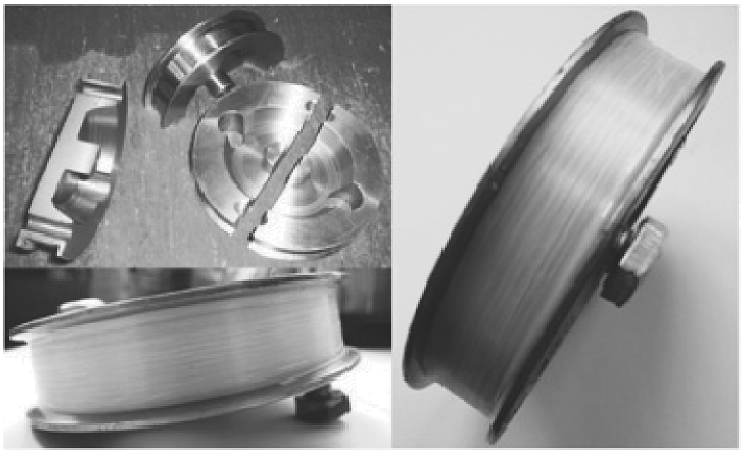
NOL rings with equipment have been assembled on testing machine MTS810. The samples have been elongated with the speed of 3 mm/min till rapture. Exemplary engineering stress-strain curve is presented in Fig. 2. Arithmetic mean taken from 10 raptured samples equals to 30,46 kN with standard deviation 1.69.

Static and fatigue tests have been done using hydraulic pulsator MTS 810 with rubber as an working medium (exerting pressure). Four tests samples were prepared with diameter equal to 30 mm, height equal to 90 mm and thickness of the wall equal to 1,5 mm. Additionally, prepared samples were protected by band at the ends in order to limit the influence of boundary effect from the end side of the tube. Inside the sample, there were placed a pile of rubber rings covered with talc (to minimize the impact of friction to obtained results). The height of pile was equal to 80 mm (before loading). With the use of two steel pistons, fitted to fabricated samples and put inside the ends of the tube filled with rubber, the rubber inside were axially compressed.

During investigations, the quantity of pistons displacement and increase of compression load have been registered. During static loading, loads over 1 kN were achieved, but in this case irreversible effects occurred, that is fibres disconnected from each other resulting in silicone cracking. However, Failure of test sample have not occurred. While loading with compression force equal to 300 N no failure of the polymer - silicone were observed (Fig. 3a).

Due to the lack of the literature data pertaining to mechanical properties of human oesophagus, these quantities have been determined experimentally during static tensile test of piggy oesophagi. Those researches have been carried out on testing machine (ZEISS) placed in Institute of Engineering Materials and Biomaterials, Silesian University of Technology. Investigations have been done for previously estimated minimum number of samples (by the method applied in the situations where the sample size is small, thus is number of samples lower than 20). Arithmetic mean taken from eight tensile measurements equals to 118.7 N, with standard derivation 16.85. Destined tissues of piggy oesophagi for investigations are characterized with mechanical properties similar to the human ones. On the basis of these researches, it is assumed that in human oesophagus, loading can not be higher than 200 N. For that reason, the samples have been subjected to fatigue tests in the range of 0 - 200 N, with frequency of 1 Hz. After 1000 cycles of tests, on failures of samples have been observed (Fig. 3b).

a)



b)

Fig. 1. The aramid-silicone composite materials in the tests with the non-standard NOL rings and hydraulic pulsator MTS 810 use; a) the type of non-standard NOL tooling of wound aramid fibre; b) sample installed in the grips of the testing machine

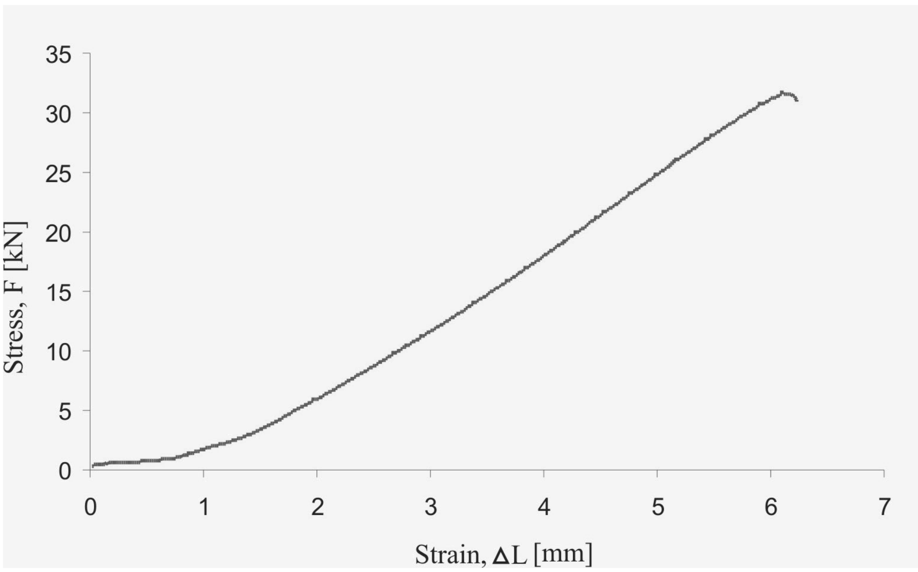


Fig. 2. Exemplary engineering stress-strain curve of the NOL rings sample

a) b)

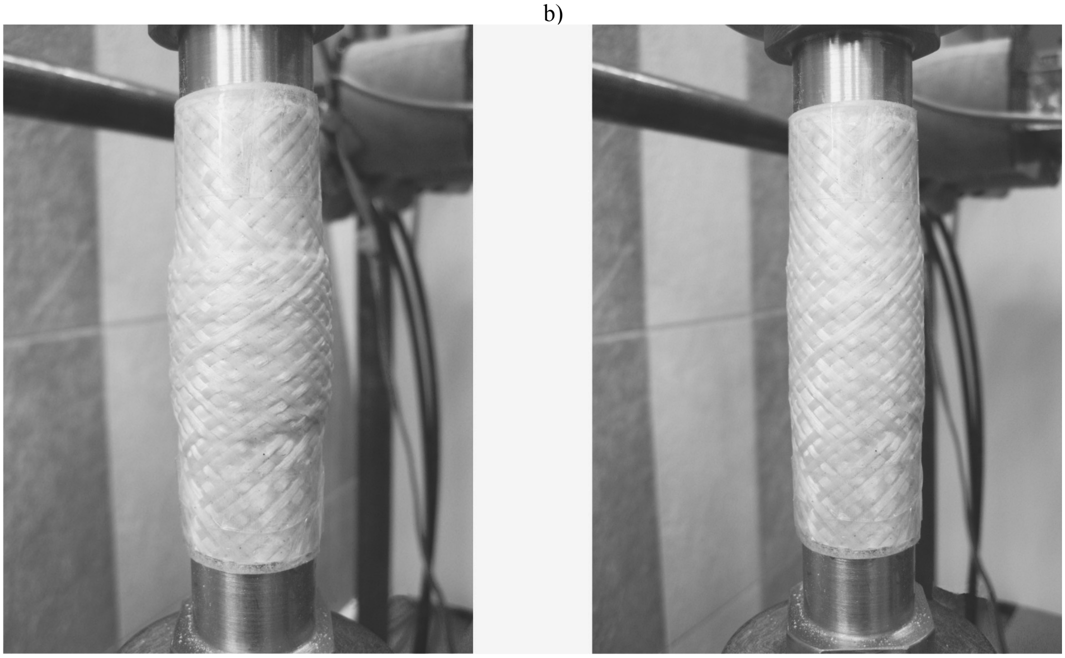


Fig. 3. The tubular sample during fatigue tests of a) static load 300N b) dynamic after 1000 cycles

Carried out pressure measurements are the starting point for further planned investigations on tubular samples with diversified fraction of reinforcing phase.

Evaluation of fabricated composite materials was done taking into consideration following criteria: organoleptic, estimation of external surface of the sample and it transparency, estimation of producibility of the fabrication process, repeatability and unit cost of fabricated sample. It has been done evaluation and comparison of selected types of warp rolls in respect of their future applications in medicine as internal oesophageal prosthesis.

5. Summary

Proposed research methods (non-standard NOL rings and tests using axial compressed rubber) can be applied in evaluation of fabricated long-fibrous material, e.g. determination of degree of silicone material binding.

During interpretation of pressure tests results according to the proposed method, it should be paid attention to the state of stresses in the wall of investigated tubular element. Ignoring fractional resistance of the rubber with sample wall, it can be obtained uniaxial stress in circumferential direction [14].

On the basis of carried out researches and observations it can be stated, that obtained tubular elements withstands assumed loads - internal pressure induced by the force of 200N.

Comparable method of investigations, consisting in compression of rubber inside the tubular sample, is original and can be successfully used as reliable alternative for commonly used research methods of tubular elements.

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