

**Krzysztof Majerski, Barbara Surowska, Jarosław Bieniaś**

Lublin University of Technology, Faculty of Mechanical Engineering, Department of Materials Science and Engineering  
, ul. Nadbystrzycka 36, 20-618 Lublin, Poland

\* Corresponding author. E-mail: krzysztof.majerski@pollub.edu.pl

Received (Otrzymano) 15.02.2012

## TENSILE PROPERTIES OF CARBON FIBER/EPOXY LAMINATES AT LOW AND ROOM TEMPERATURES

Polymer composites based on carbon fibers are used in a large number of applications in the environment of low temperatures. The current use of such composites is not limited to support structures but carbon/epoxy materials are also used successfully as primary structures in aeronautic applications. The paper presents the tensile properties of a AS7J carbon/epoxy laminate at low and room temperatures. The composite prepreg system includes epoxy M12 resin as the matrix and unidirectional high-strength carbon fibers as reinforcement. Tensile tests were performed at room temperature (RT), 223 and 153 K using an MTS 322.31 testing machine equipped with an environmental chamber. The strain gauge Vishay CEA-06-125UT-350 was employed to measure the strain. The tensile modulus, strength and Poisson ratio at different temperatures were compared. The failure analysis of the samples was investigated by scanning electron microscopy (SEM). The fracture morphology at the interface between the fiber and matrix was also observed. The results have shown that the mechanical properties depend on temperature. The tensile modulus of the AS7J carbon/epoxy laminate increased as the temperature decreased, however, the tensile strength showed a slight decrease at lower temperatures. The value of Poisson's ratio fell slightly when the temperature decreased. The failure analysis of the specimens indicates that the nature of the destruction of the samples is also dependent on temperature. Classic morphology damage known as high-energy damage was observed in samples tested at room temperature and a more complex type of damage occurred in the samples at the temperature of 153 K.

**Keywords:** carbon fibre, laminates, tensile property

## WYTRZYMAŁOŚĆ NA ROZCIĄGANIE LAMINATÓW O OSNOWIE ŻYWICY EPOKSYDOWEJ WZMACNIANYCH WŁÓKNAMI WĘGLOWYMI W WARUNKACH TEMPERATUR NISKIEJ I POKOJOWEJ

Laminaty polimerowe zawierające włókna węglowe są często stosowane w środowisku niskich temperatur. Obecnie wykorzystanie tego typu materiałów nie ogranicza się do budowy struktur pomocniczych, ale laminaty na osnowie żywicy epoksydowych wzmocnionych włóknami węglowymi są z powodzeniem stosowane na główne elementy konstrukcyjne w przemyśle kosmicznym. W pracy przedstawiono wyniki badań wytrzymałości na rozciąganie laminatu wzmocnianego włóknami AS7J na osnowie żywicy epoksydowej w różnych temperaturach. Materiał badawczy składał się z warstw jednokierunkowej taśmy prepregowej zawierającej wysokowytrzymałe włókna węglowe o osnowie żywicy epoksydowej M12. Badanie wytrzymałości na rozciąganie przeprowadzono w temperaturach 295, 223 oraz 153 K za pomocą maszyny wytrzymałościowej MTS 322.31 wyposażonej w komorę środowiskową. Do wyznaczenia wartości odkształceń wykorzystano tensometri oporowe Vishay CEA-06-125UT-350. Dokonano porównania wartości modułów sprężystości wzdłużnej, wytrzymałości na rozciąganie oraz współczynnika Poissona uzyskanych w różnych temperaturach. Analizy zniszczenia próbek dokonano przy pomocy elektronowego mikroskopu skaningowego. Przeprowadzono obserwacje morfologii przelomów oraz granicy rozdziału włókno-osnowa. Badania dowiodły, że badane właściwości mechaniczne są zależne od temperatury. Wartość modułu Younga wzrosła wraz ze spadkiem temperatury, natomiast wytrzymałość na rozciąganie okazała się niższa w obniżonej temperaturze. Wartość współczynnika Poissona spadła nieznacznie wraz ze spadkiem temperatury. Analiza fraktograficzna wskazuje, że charakter zniszczenia próbek również zależy od temperatury. Zaobserwowano klasyczną morfologię zniszczenia określaną jako wysokoenergetyczną dla próbek badanych w temperaturze pokojowej oraz bardziej złożony typ zniszczenia dla próbek badanych w temperaturze 153 K.

**Słowa kluczowe:** włókna węglowe, laminat, właściwości wytrzymałościowe

## INTRODUCTION

Fiber-reinforced polymer composites are used in large numbers of applications at low temperatures [1, 2]. Because of their exceptional mechanical properties, CFRP composites (Carbon Fiber Reinforcement Poly-

mer) confirmed their suitability for use in many demanding applications. The mechanical properties of these materials can be shaped according to need [3, 4]. There is a great deal of research work on the tensile

strength of CFRP composites at room temperature [5-7]; however, information on the behavior of these composites at low temperatures is not complete. There are reports of changes of the mechanical properties at cryogenic temperatures and the consequences of the use of thermo-mechanical cycles [8]. Due to the diversity of composite systems, it is necessary to attain data on the impact of low temperatures on the tensile strength and modules of carbon fiber/epoxy laminates. Moreover fractographic analysis is an important tool for a complete understanding the nature and character of failure in composite materials [9].

The paper presents the study of tensile properties of a carbon fiber/epoxy laminate at low and room temperatures.

## MATERIALS AND METHODS

A unidirectional laminate was prepared using 8 layers of UD134 AS7J 12K/M12 carbon fiber/epoxy prepreg with a 35 wt.% nominal resin content (Hexcel, USA). The laminates were produced by the autoclave technique in accordance with the manufacture's recommendation. After curing, the laminate was cut into samples according to ASTM D 3039. The specimen tabs were bonded with a thin adhesive film with low-temperature resistance.

The environmental chamber is part of the MTS 322.31 testing machine. The chamber was cooled by liquid nitrogen, delivered to the chamber from a pressure vessel (Cryotherm). The strain measurement was realized by Vishay CEA-06-125UT-350 single strain gauges. The load and strains were recorded using a Hottinger Baldwin Messtechnik MGCplus AB12 considering compensation for induced thermal strains of the gauges at low temperatures. The samples were fixed in mechanical wedge grips in which the inserts were pre-screwed with four screws each. The sample was placed in a climatic chamber, which underwent sweetening to temperatures of 225 and 153 K. The test was initiated with a delay of 15 minutes to stabilize the temperature inside the chamber. The test was conducted at a constant speed of 2 mm/min until breaking of the sample. Data on the measuring force, displacement of the actuator and the data from the strain gauges were recorded at a frequency of 10 Hz. To prevent sliding of a specimen from the grips, a preload of 5 kN was applied. The tensile properties were determined according to ASTM D 3039.

## RESULT AND DISCUSSION

### Tensile properties

The tensile modulus and strength of the UD laminate with the value of standard deviation are shown in Figure 1. The results are based on upon the arithmetic average of six testing specimens. It was noted that the

investigated mechanical properties depend on temperature. The basic parameter which is the tensile strength decreased about 7% at 223 K compared to RT and about 8% at 153 K. These results correspond to [8] which also received a tensile strength decrease with decreasing temperature while [10] shows an increase in tensile strength of a unidirectional carbon/epoxy laminate at low temperatures. The decrease in tensile strength may be caused by several factors such as a brittle matrix or an increase in residual stress in the composite material. Lowering the temperature increases the size of the fibers in the radial direction and shortening in the direction of the longitudinal axis, while the matrix expands in all directions [11-13].

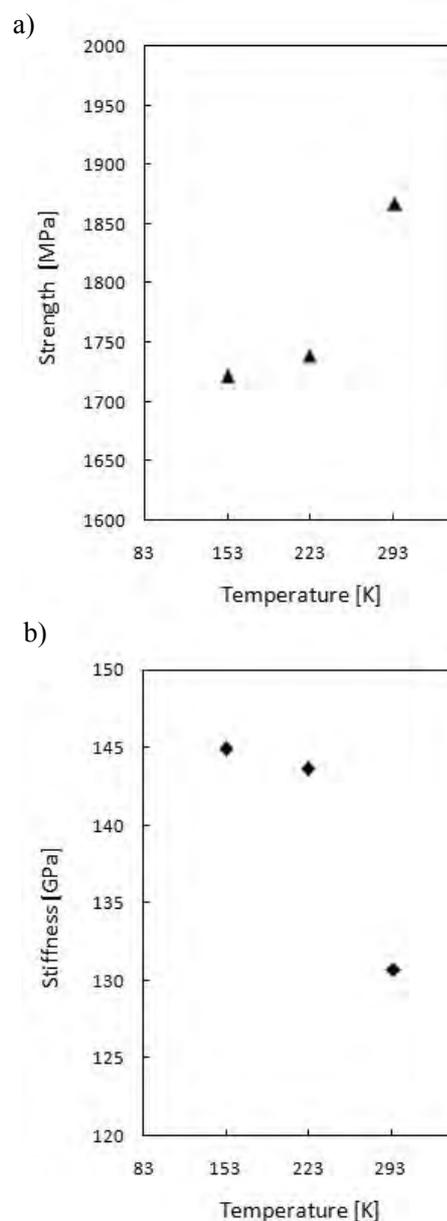


Fig. 1. Results of: a) tensile strength and b) stiffness of M12/35%/UD134/AS7/300 unidirectional specimen as a function of temperature

Rys. 1. Wyniki badań: a) wytrzymałości na rozciąganie i b) sztywności próbek z jednokierunkowego laminatu M12/35%/UD134/AS7/300 w funkcji temperatury

The tensile modulus increases as the temperature decreases but the increase between 153 and 223 K was smaller than 223÷295 K which amounted to 9%. These results correspond to [8] and an explanation of this phenomenon is probably a sharp increase in fiber brittleness in this temperature range. The value of Poisson's ratio (Fig. 2) did not change much. This value decreased slightly with a decrease in temperature.

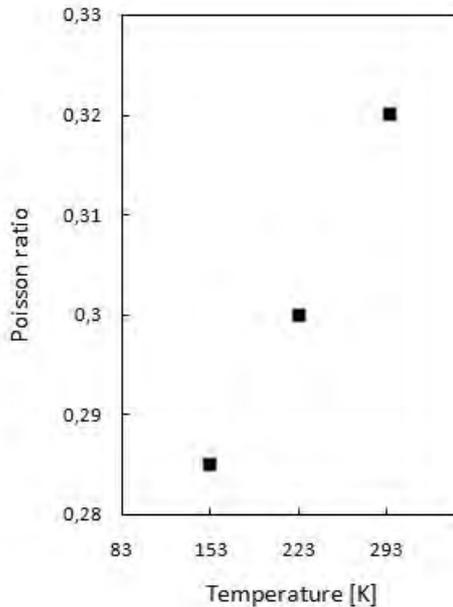


Fig. 2. Poisson ratio of M12/35%/UD134/AS7/300 unidirectional specimen as function of temperature

Rys. 2. Współczynnik Poissona próbek z jednokierunkowego laminatu M12/35%/UD134/AS7/300 w funkcji temperatury

### Fractographic analysis

Analysis of the fracture was carried out for samples tested at two extreme temperatures (295, 153 K). The data of the specimens that failed in the tensile test are plotted in Figure 3. These samples were chosen as representatives of each group. The fracture in both cases differs significantly. The sample tested at RT has almost split fibers and was separated into multiple pieces. On the other hand, the sample tested at 153 K was destroyed in one place across the axis of the sample. A significant difference in the morphology of failure has probably been linked to the brittleness both the

matrix and fibers at low temperatures. Similar results were obtained in [8], however, Kim et al. did not receive such a significant difference in the nature of the failure of the samples at different temperatures. This is probably due to the subtle differences in used in the prepreg systems. There are many species of epoxy resins used to produce prepreg systems based on high strength carbon fibers, which have different characteristics of mechanical properties, which can result in a difference in the fracture morphology of the laminates.

The fractured surfaces of the specimens was investigated by SEM. Figure 4a shows the fracture surface morphology of the sample tested at RT while Figure 4b presents the fracture surface morphology of the sample tested at 153 K. The difference in the morphology of these fractures lies in the fact that the filament bundles in samples tested at RT are located at different heights and it is recognized as the high energy fracture region, while in the case of the sample tested at low temperature, the filament bundles are in one plane. The high energy morphology is typical for tensile tests of unidirectional carbon/epoxy laminates at RT [14]. The morphology of low-energy fracture is identified with the failure of the laminate by the compressive load; however, this phenomenon also occurs in tensile tests. The explanation for this phenomenon is given by S. Greenhalgh in [15]: '... the compressive recoil from the initial tensile failure can be of a dry severity that the dynamic stress wave passing down the specimen compressive exceeds the strength ...'. In the case of the morphology of sample 4b, this explanation is not entirely accurate because, as shown in Figure 3, there is only one breakthrough throughout the sample and the SEM observations showed that the morphology of the low energy is the dominant form of failure in the case of the samples tested at 153 K.

Another point of discussion is the bond between the fibers and matrix. In Figure 4a, the connection of the fiber and the matrix is correct while the morphology of fracture 4b shows signs of debonding among the fibers. It is believed that the residual stresses induced in the composite by the mismatch of the coefficient of thermal expansion at low temperatures results in micro-cracks and therefore weakens the bond between the fibers and matrix.

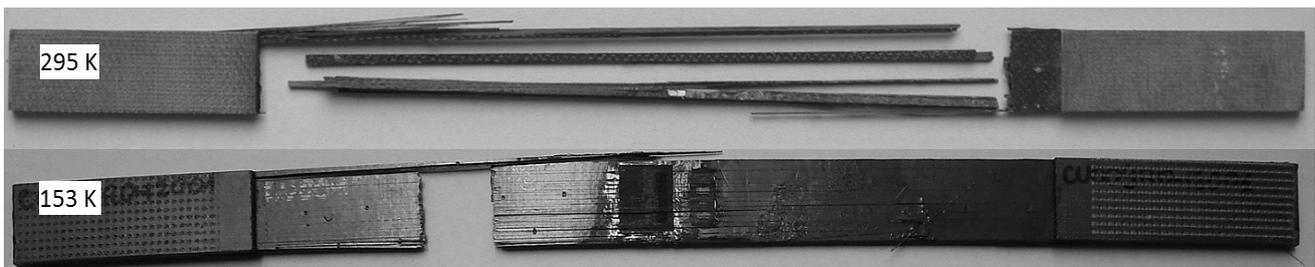
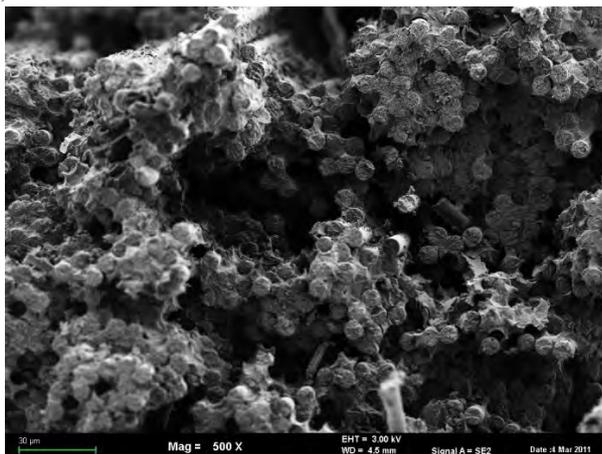


Fig. 3. Failure types of specimens destroyed in tensile tests at 295 and 153 K

Rys. 3. Rodzaje zniszczenia próbek występujące w próbie rozciągania w temperaturach 295 i 153 K

a)



b)

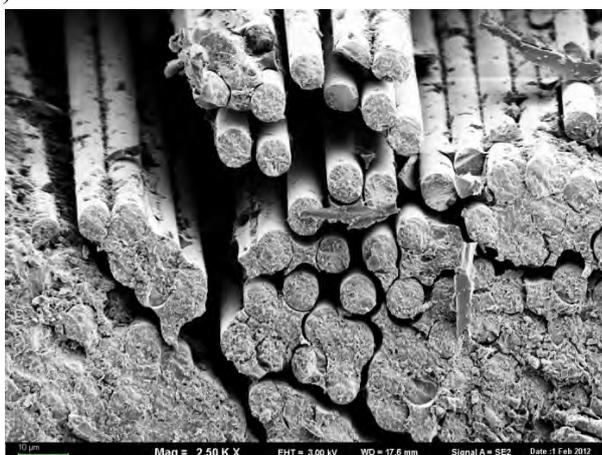


Fig. 4. SEM image of fracture surface morphology of samples tested (a) at RT, (b) at 153 K

Rys. 4. Mikrofotografia SEM morfologii przelomu próbek badanych w temperaturze (a) pokojowej, (b) 153 K

## CONCLUSIONS

In this study the temperature dependence of the tensile properties and fractographic analysis of a unidirectional carbon fiber/epoxy laminate was investigated. It was found that the tensile strength of the considered laminate decreases with decreasing temperature. At 153 K the composite stiffness is much greater than at room temperature. The good quality and correctness of the manufacture of the test materials was confirmed by SEM analysis. Fractographic investigation confirms the results of the strength tests and explains the nature of the phenomena occurring in the process of failure. The presence of high energy fracture in samples tested at RT, and low energy in the samples tested at 153 K have been found. The fracture morphology of the samples tested at low temperatures showed more cracks on the fiber-matrix boundary during microscopic examination. The detachment of fibers from the resin observed

at low temperatures is a phenomenon whose cause is the mismatch of the thermal expansion coefficient between the components, which causes micro cracks in the matrix structure and the weakening bond at the interface of the fiber and matrix. Under the conditions in which the tests were performed, the resin has a high rigidity and is not capable of plastic deformation which results in the rapid growth and joining of cracks running parallel to the fibers at the moment of breaking of the sample.

## Acknowledgment

*Financial support of the Structural Funds in the Operational Programme - Innovative Economy (IE OP) financed from the European Regional Development Fund - Project No POIG.0101.02-00-015/08 is gratefully acknowledged.*

## REFERENCES

- [1] Willis P., Hsieh C.H., Space Applications of Polymeric Materials, Society of Polymer Science - Kobunshi Japan 1999.
- [2] MIL-HDBK-17-1F, Composite Materials Handbook, 2002.
- [3] Schutz J.B., Properties of composite materials for cryogenic applications, *Cryogenics* 1998, 38, (1), 3-12.
- [4] Freeman W.T., The use of composites in aircraft primary structure, *Comp. Eng* 1993, 3, 767-775.
- [5] Miracle D.P., Donaldson S.L. (eds), *ASM Handbook Vol. 21 Composites*, ASM International, 2001, 1875-1900.
- [6] Morgan P., *Carbon Fibers and their Composites*, Taylor and Francis Group, New York 2005.
- [7] Campbell F.C., *Manufacturing Technology for Aerospace Structural Materials*, Elsevier, London 2006.
- [8] Kim M., Kang S., Kim CH., Kong Ch., Tensile response of graphite/epoxy composites at low temperatures, *Composite structures* 2007, 79, (1), 84-89.
- [9] Greenhalgh E.S., Hiley M.J., Fractography of polymer composites current status and future issues, In: *Proceedings of the 13th European Conference on Composite Materials (ECCM13)*, Sweden, Stockholm 2008.
- [10] Reed R.P., Golda M., Cryogenic properties of unidirectional composites, *Cryogenics* 1994, 34, (11), 909-928.
- [11] Timmerman J.F., and the others, Matrix and fiber influences on the cryogenic microcracking of carbon fiber/epoxy composites, *Composites Part A* 2002, 33, 323-329.
- [12] Mallick P.K., *Fiber-reinforced Composites: Materials, Manufacturing, and Design*, Marcel Dekker, New York 1993.
- [13] Surendra Kumar M., Sharma N., Ray B.C., Mechanical behavior of glass/epoxy composites at liquid nitrogen Temperature, *Journal of Reinforced Plastics and Composites* 2008, 27, (9), 937-944.
- [14] Clements L.L., Fractography of unidirectional graphite-epoxy as a function of moisture, temperature, and specimen quality, *Journal of Materials Science* 1986, 21, (6), 1853-1862.
- [15] Greenhalgh E., *Failure Analysis and Fractography of Polymer Composites*, 1 edition 2009.