Polish Society of Composite Materials
Polskie Towarzystwo Materiałów Kompozytowych

20: 2 (2020) 78-84

COMPOSITES THEORY AND PRACTICE
ISSN: 2084-6096
ISSN (online): 2299-128X

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Received (Otrzymano) 19.05.2020

METHODOLOGY FOR QUANTITATIVE ASSESSMENT OF BASALT FIBERS OBTAINED IN CONTINUOUS PROCESS

In 2019, the first basalt fiber production line was created in Poland. The fiber is produced in a continuous process, according to the technology developed by Polski Bazalt S.A. In order to assess the microstructure of the manufactured product, a number of tests were carried out, according to previously developed procedures. The presented results relate to the study of the basalt fiber microstructure using light microscopy, electron microscopy and atomic forces. The research was aimed at characterizing the fibers, but also developing research procedures that allow assessment of the basic fiber parameters under post-production conditions. The research was conducted in the field of quantitative and qualitative assessment of the basalt fiber microstructure, its diameter, and the size distribution of this value. In addition, attempts were made to assess the thickness of the sizing (as an impregnation layer) on the fibers obtained employing different parameters of the drawing process and various types of impregnation. Based on the obtained results, measurement and research procedures were implemented in the quality control system of the Polski Bazalt company. Tests carried out as part of these procedures confirm the repeatability in terms of the quality and diameter of the produced fiber.

Keywords: basalt fiber, microstructure, SEM, sizing thickness

INTRODUCTION

Basalt fibers are a product made only of natural volcanic rocks. The process of producing such fibers is similar to glass fibers, but requires higher process temperatures of 1300÷1550°C. It is necessary due to the chemical and crystallographic composition of basalt rocks [1]. Basalt fibers, like other technical fibers, must be covered by sizing in the final production process. The fibers then obtain the expected technical and process parameters for use in composites [2-4]. Basalt fiber is characterized by high strength properties, resistance to high and low temperatures, as well as acid and alkaline environments [5]. Basalt fibers are also resistant to abrasion, moisture and weather conditions. The advantage of basalt fiber as a reinforcement of composites is based on the following features [1, 6]:

- High strength (2.5 times greater than alloy steel and 1.5 times that of glass fibers) [2].
- High temperature stability in the range of –200°C to 600°C.
- Can be used with a variety of materials such as plastics, adhesives, or resins.
- Low density 2.7 g/cm^3.
- High chemical resistance.

In addition, basalt fiber is resistant to UV radiation, does not melt and does not plasticize under the influence of variable temperature, has good adhesive properties to resin matrices, is an electrical and thermal insulator and is transparent to a magnetic field [7]. From basalt fibers we can obtain roving and various forms of fabrics such as: directional fabrics (used in composites as reinforcement), multiaxial fabrics (used in composites as reinforcement), sealants (used in thermal insulation), meshes (used in filter applications, as protective meshes or road and construction) and staple fiber (as distributed reinforcement of composites and concrete). Due to their properties, basalt fibers are a substitute for commercially used fibers, i.e. glass, aramid, or carbon [8].

Basalt fibers are widely studied in science and used in technology as a perspective material due to their natural origin and renewal, for example in the matrix of biodegradable composites [9]. Basalt fiber is currently used in the form of composites wherever it can replace steel products, obtaining higher strength parameters and full resistance to external conditions without exposure to corrosion in sectors such as automotive, maritime, construction (roads, bridges, sea fortifications, mine shafts, pipelines) and in specialized applications [10, 11].

In order to be able to properly assess the mechanical parameters of basalt fiber, it is necessary to develop appropriate research techniques both for dry fiber/ roving [12] and for finished composite products [13].

The article presents and compares the research techniques developed by Polski Bazalt S.A. on the first basalt fiber produced in Poland.

RESEARCH METHODOLOGY

Metallographic samples were prepared from cross--sections of bundles and roving as well as cross-sections of longitudinal sections of fiber. Preparation of the metallographic samples consisted in taking fiber sections and roving and embedding them in resin using a mounting press. The cylinder obtained in this way was then cut with an automatic saw with a diamond blade. Another sample preparation technique was to use special grips to place the roving in a press chamber. The samples with the conductive resin were obtained by pressing under hot pressure, at the temperature of 160°C. On the other hand, the samples prepared with non-conductive resin were obtained at 120°C. The next steps involved grinding and polishing the surface of the samples. The obtained samples were not etched. Figure 1 presents three examples of samples to examine the cross-sectional area of both the types of resins and a sample with sections to observe the fiber surface and the longitudinal cross-section of the fiber. Metallographic samples were produced by embedding four bundles of basalt fibers in both types of resins. Disposable metallographic handles were used to obtain vertical fiber orientation in the fiber cross-section investigations.

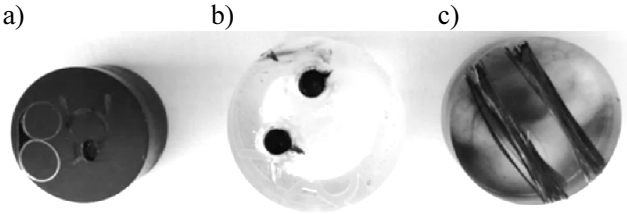

Fig. 1. Samples prepared for microscopic examination of basalt roving: a) cross-section of conductive resin, b) cross-section of non-conductive resin, c) longitudinal section of non-conductive resin

The microstructure and the distribution of the basalt fiber diameters were studied, along with the measurement of the thickness distribution of the impregnate strengthening the raw basalt fibers (sizing) using the following microscopic techniques:

- Light microscopy. Investigations were performed on two types of light microscopes (KEYENCE, NIKON) with magnification up to 1000x.
- Scanning electron microscopy (SEM). These examinations were conducted on five types of microscopes differing in parameters and source of electron emission. This allowed assessment ofthe impact of various microscope configurations on the image quality, its resolution and zooming options to optimize the examination of the basal fibers. The investigations were carried out using:
  - HITACHI 3500 microscope with a resolution of up to 0.4 nm at magnifications from 15 to 300,000x. It is equipped with SE and BSE detectors and an EDS X-ray spectrometer.
  - PHENOM PRO integrated with an EDS energy dispersion spectrometer. It has magnification of 20 to 150,000x, with a CeB_6 electron source.
  - HITACHI TM 1000 with magnification of 20 to 10,000x with a tungsten cathode.
  - VEGA (TESCAN company) with a tungsten cathode
  - QUATTRO from Thermo Scientific, with field emission, SE and BSE detector.

The studies included qualitative microstructure analysis with quantitative dimension analysis. Observations of the microstructure were made on a set of samples which included specimens of single fiber sections and roving, both longitudinal and cross-sectional. These examinations concern measurement of the fiber diameters and the size distribution of these diameters throughout the entire roving. In addition, investigations are planned to assess the thickness of the sizing on individual fibers and to assess the uniformity of this thickness around the perimeter of the fiber.

RESULTS – LIGHT MICROSCOPY

Example examination results are shown in Figure 2, where micrographs obtained of the samples with cross-sections of the fibers in the roving and on the longitudinal sections of the fibers are presented. The micrographs were taken on a KEYENCE VHX-600 microscope. The diameter of the selected fibers was measured, but due to the shallow depth of field, the results not exactly accurate.

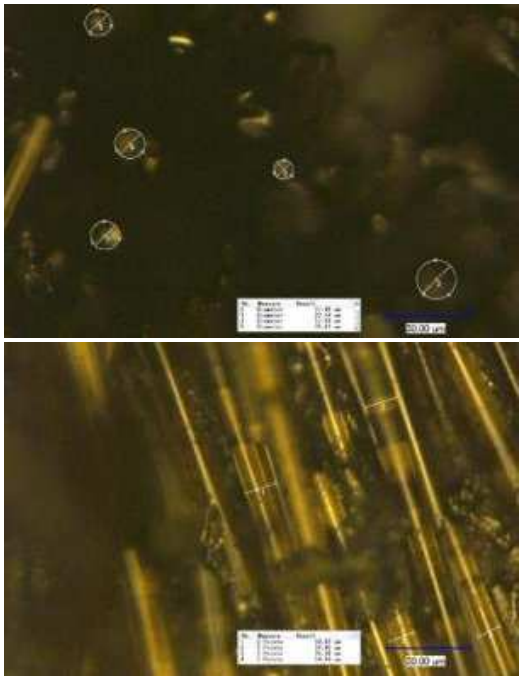

Fig. 2. Image of basalt fiber microstructure on cross-sections and on longitudinal sections of fibers

The results obtained using the NIKON microscope were worse than the micrographs from the KEYENCE microscope. The too shallow depth of field and small magnification do not allow reliable results of the fiber diameters to be obtained.

RESULTS – SEM

HITACHI TM-1000

Figure 3 shows micrographs of the fiber microstructure on the cross-section of the sample included in the two types of resin. The micrographs were taken using high energy electrons (BSE mode) due to the non-inductive nature of the fiber itself. The phase contrast of the micrographs does not allow identification of the sizing layer.

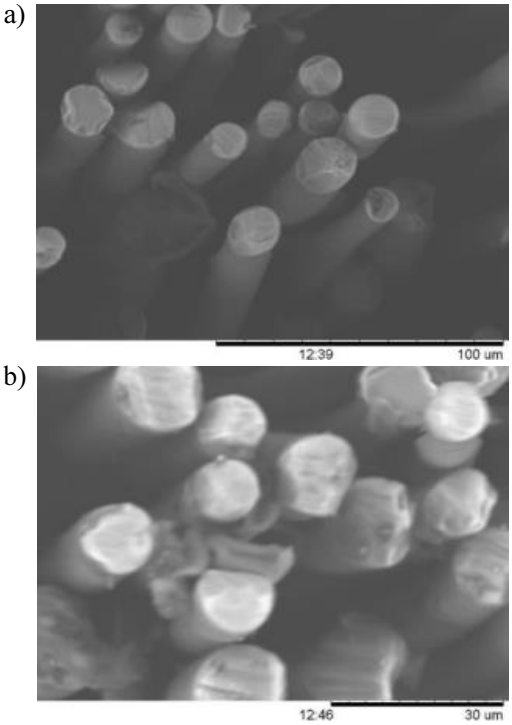

Fig. 3. Micrographs of fibers in roving at 1000x magnification, sample in non-conductive resin (a) and in conductive resin (b)

PHENOM PRO

Examinations using the PHENOM microscope were performed on a sample prepared with the non-conductive resin. A conductive layer was used here to discharge the charge from the surface of the sample. Figures 4-6 show micrographs with varying degrees of magnification obtained in the secondary electron and backscattered electron modes. The micrographs were obtained at a magnification of 4000x to 9000x.

The micrographs obtained in the SE mode are characterized by high resolution and contrast allowing observation of cleavage planes on the surface of the fiber cross-sections. Figures 6 and 7 show micrographs with registered diameter measurements for selected fibers and attempts to measure the thickness of the sizing layer on the fiber.

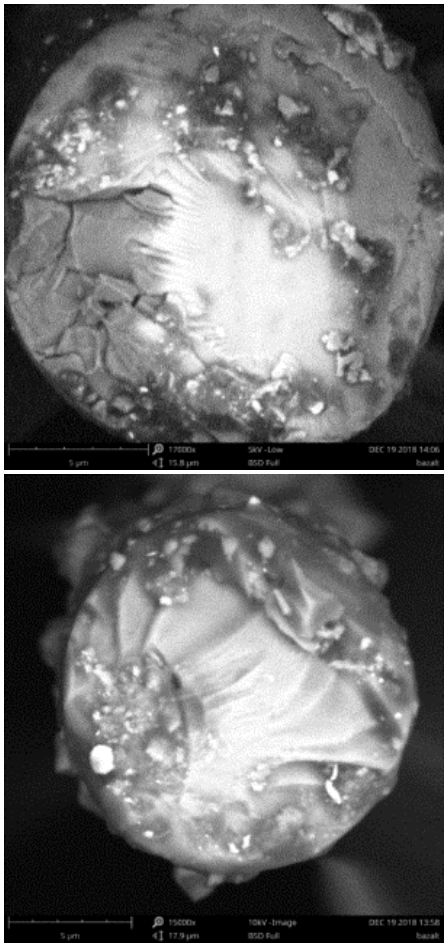

Fig. 4. Image of basalt roving microstructure at 9000x magnification

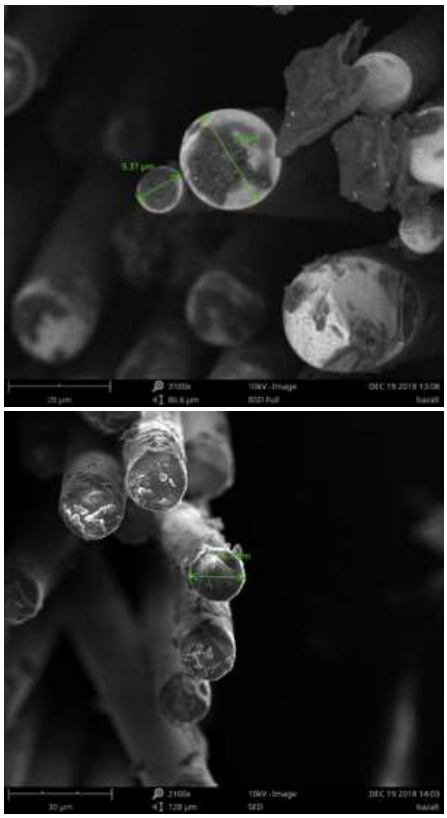

Fig. 5. Micrographs of basalt roving microstructure with diameter measurements

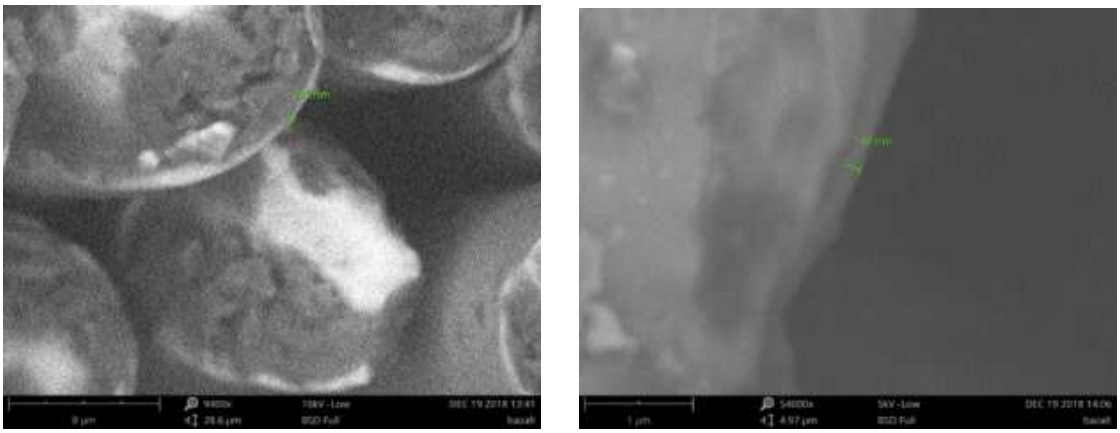

Fig. 6. Micrographs of basalt fiber microstructure at 9000x magnification with attempt to assess thickness of sizing layer

HITACHI 3500

The investigations were carried out on a sample with a cross-section of a fiber embedded in the conductive resin. However, due to the strong loading of basalt fibers alone (Fig. 7), only observations using the BSE detector were performed.

The BSE mode limits the possibility of obtaining a good topographic contrast. Nevertheless, assessment of the homogeneity of the grain size (diameter) distribution is possible.

The diameters of a dozen or so fibers were measured based on the micrographs of the fiber microstructure obtained by means of the four different microscopic techniques. The measurement was performed using the proprietary software of the KEYENCE, PHENOM and TM 1000 microscopes (Hitachi). The results are presented in the diagram presented in Figure 8.

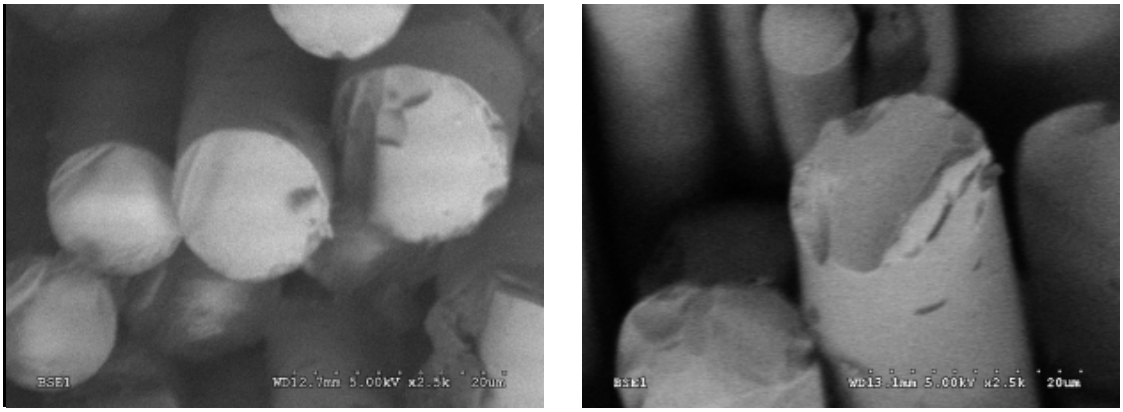

Fig. 7. Micrographs of fiber microstructure at magnifications in range of 1000-6000x

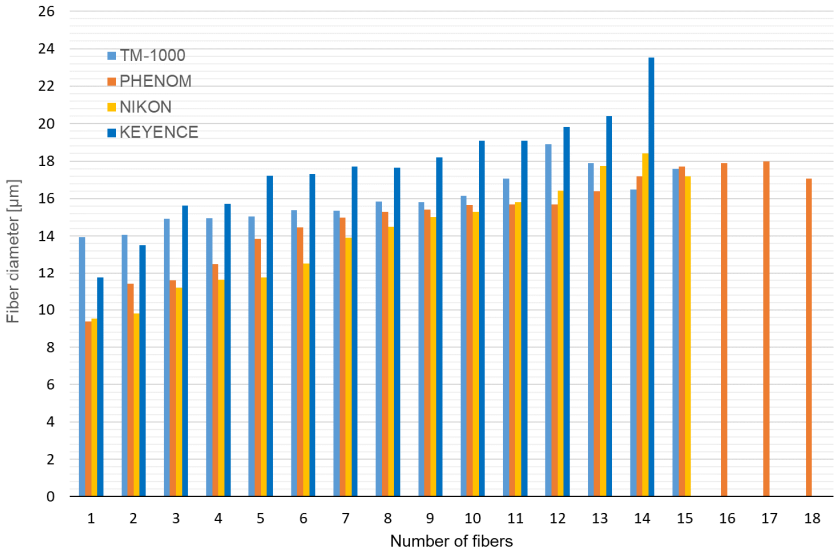

Fig. 8. Measurement results of dozen or so fibers, obtained on various types of microscopes

Assessment of sizing layer thickness

The investigation was carried out using a QUATTRO microscope made by TERMO FISHER with an electron source in the form of a Schottky type field emission gun. Micrographs of samples prepared with scissor-cut roving are shown in Figure 9. They confirm the size of the fiber, but identification of the sizing layer is difficult.

The possibilities of the microscope allowed frames to be found giving the chance to approximately estimate the thickness of the sizing layer on the surface of the studied basalt fiber, which is confirmed by the micrographs in Figure 10.

Due to the difficulty of identifying the sizing layer and assessing its thickness, a trial series of samples was made using an ion polisher. Thanks to the samples prepared in this way, the obtained micrographs allowed not only the microstructure of the fiber and its diameter to be assessed, as shown in Figure 10, but also the thickness of the impregnation layer to be measured (Fig. 11). The observations were carried out in the low vacuum mode due to the non-conductive nature of the preparation and due to the strong "drift" of fibers under the influence of high vacuum.

A double layer can be observed on the surface of the fiber. The external one was formed as a result of the deposition of basalt particles removed from the cross-sectional surface with an ion beam during polishing. "Redeposition" of the basalt particles on the polymer layer protects it against the polishing ions. Nonetheless, it creates some problems in interpretation of the obtained results. Figure 12 shows the results of measuring the sizing layer in several places of selected fibers on the cross-section of the roving.

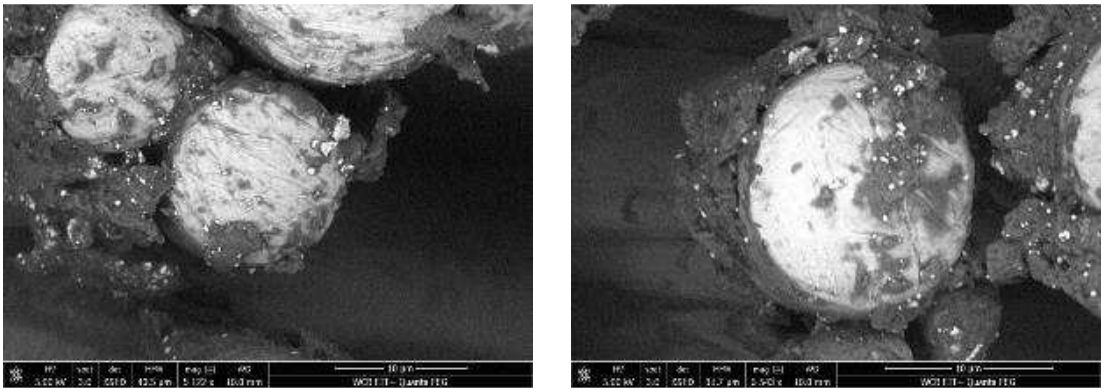

Fig. 9. Cross-sections of individual basalt fibers

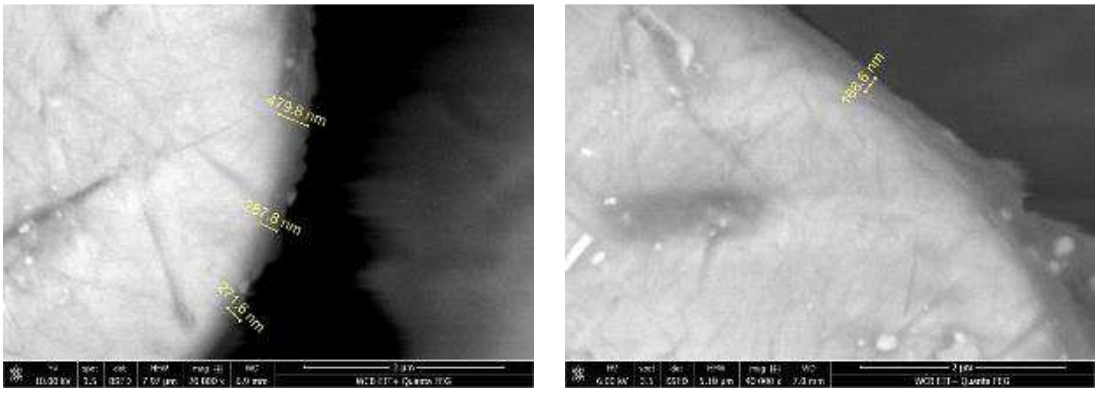

Fig. 10. Fragments of cross-sections of fibers with identified sizing layer

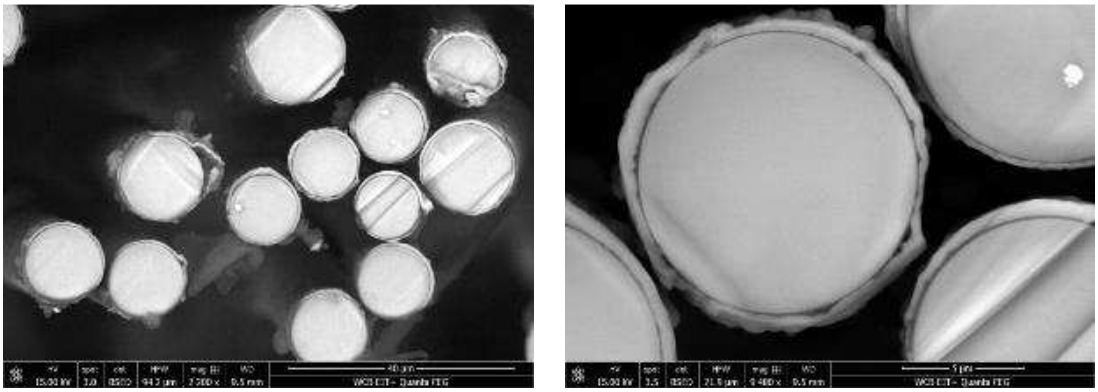

Fig. 11. Micrographs of basalt fiber cross-sections, with visible layer of impregnation, obtained on roving treated by ion polishing

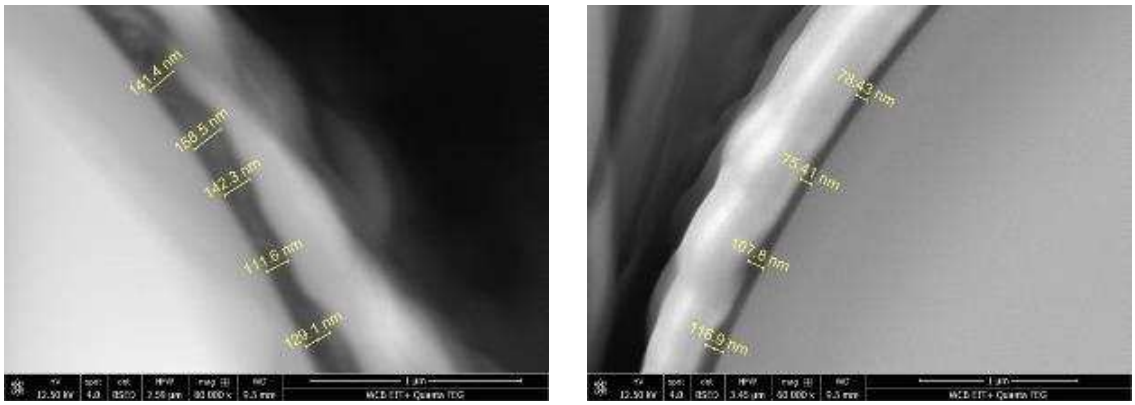

Fig. 12. Results of measuring sizing layer on fragments of fiber cross-sections

The results obtained on mechanically cut fiber and on two ion-polished fibers are presented in a graph (Fig. 13).

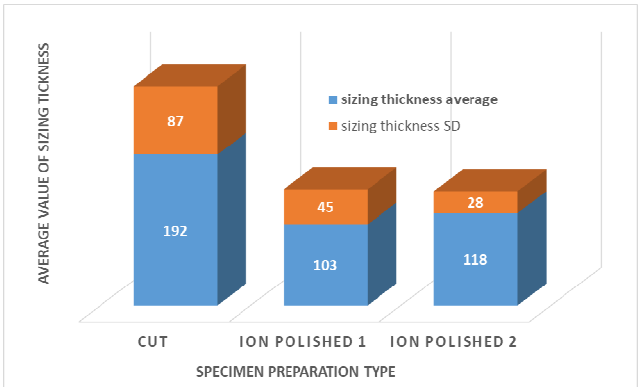

Fig. 13. Sizing thickness for fibers prepared by different techniques

Chemical composition characterization was also carried out using an EDS analyzer. The results, in the form of mapping of the elements found in basalt rock as well as in the polymer preparation, confirm the identification of the layer where the concentration of carbon elements from the organic sizing is visible (Fig. 14).

Based on the qualitative assessment of the distribution of elements in the sizing, it is also possible to estimate its thickness as a confirmation of the measurements.

CONCLUSIONS

1. Light microscopy of both cross-sections and longitudinal sections of fiber sections only allow estimated assessment of the fiber diameter and size distribution.
2. Precise fiber diameter assessment using each of the SEM microscopes is possible. Among the microscopes with a thermo-emission electron source, the best results were obtained by the LaB6. There are limitations in the area of sizing thickness assessment related to image resolution above 15,000x magnification. The results of fiber diameter measurements using various microscopic techniques are summarized in Table 1. The table shows the average values from a population of approximately 15 measurements using each technique. The standard deviation value was determined for each of them. The results indicate a relatively small distribution of values, i.e. the diameter of all the fibers in the roving is similar to the average value, which for all the measurements is 15.56, and the deviation within the five techniques used is also at a similar level (3.05).

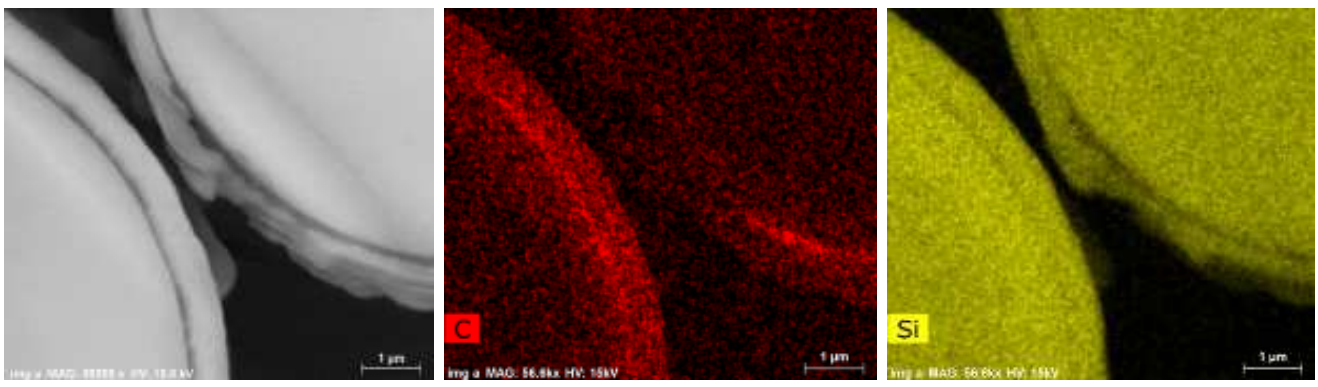

Fig. 14. Mapping of elements on cross-section of two fibers

TABLE 1. **Results of assessment of average value of fiber diameters**

Measurement device	Average value of fiber diameter	SD
KEYENCE (LM)	17.52	2.92
NIKON (LM)	13.92	3.05
HITACHI 3500 (SEM)	15.88	4.92
PHENOM (SEM)	15.83	2.87
HITACHI TM – 1000 (SEM)	16.27	2.60
AVERAGE	*15.68*	*3.20*

3. Non-conductive basalt fibers require observation in the low vacuum mode and at low beam energies, which will allow high resolution imaging. High vacuum causes the surface to charge, and a high beam current causes the fibers to deflect, making it difficult to accurately analyze the sizing thickness. In addition, polymer sizing is very sensitive to an electron beam, which forces one to work at lower voltages. The quality of the area is improved by spraying the sample with a conductive layer, which will ensure greater efficiency than the low vacuum mode,
4. Preparation of the fiber cross-sections by mechanical cutting does not allow parallelism of the received cross-sections to be obtained, making it difficult to assess the sizing thickness in a larger fiber population. In addition, it causes the polymer sizing layer to slide over the basalt, which makes it difficult to accurately examine its thickness.
5. Surface etching of the roving cross-section with the ion polisher leads to the redeposition of basalt on the surface of the polymer sizing. On the one hand, this allows the polymer to be protected against greater degradation, on the other hand, it hinders measurement of the thickness of the layer covered partially by redeposited basalt.
6. The basalt redeposition on the surface of the fibers as a result of ion polishing is confirmed by the results of chemical tests of the core. The carbon concentration in the layer area indicates the presence of an organic polymer. However, the increased share of basalt components (among others Si) on both sides of the sizing indicates the redeposition effect.
7. The thickness of the polymer sizing layer is heterogeneous; different thicknesses around the basalt core are visible, the places where the polymer does not adhere to the basalt are visible. This is not the effect of sample preparation but the technology of layer production.

Acknowledgment

The results were obtained under project no. POIR. 01.01.01-00-0802/17.

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