ENGINEERING STRUCTURES AND TECHNOLOGIES
ISSN 2029-882X / eISSN 2029-8838
2017 9(4): 207–213
doi:10.3846/2029882X.2017.1417062

Research paper

FRACTURE RESISTANCE ANALYSIS OF PEEK-POLYMER

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Received 15 November 2017; accepted 11 December 2017

Abstract. PEEK is a thermoplastic polymer with halfcrystallic structure – the scientific name of this material is poly(oxy- 1,4-phenylene-oxy-1,4-phenylenecarbonyl-1,4-phenylene). It has a high melt and glass transition temperatures (T_m = 340 °C, T_g = 143 °C), high chemical resistance and is melt processable. Thus it has been used in a variety of structural and insulation applications. Nowadays, one of them is the biomedical application. The mechanical properties of PEEK have been extensively investigated in many research papers. However, there is not so high number of papers devoted to the fracture susceptibility of PEEK-material. Therefore, the the aim of this work is to present the results of studies on PEEK material with the use of fracture toughness test and digital image correlation. In order to conduct the tests, there were used two types of samples: SENB and CT. In comparison with other polymeric materials subjected to biomedical application, PEEK material presents relatively good fracture resistance with their biocompatibility.

Keywords: PEEK material, fracture toughness, DIC, biomaterials, fractography, stress intensity factor.

Introduction

Nowadays, there are several types of materials used in the in vivo condition: metals, ceramics, polymers and composites. Most of the implants are produced from biomaterials like stainless steel or nickel and titan alloy. Those materials have good mechanical strength, scrub resistance and non-toxic properties. However, metal biomaterials have some properties which are particularly undesirable in biomedical engineering. Spinal fusion operations are more and more popular on the whole earth. There were about 100 000 those operations performed in USA. In 1995 there were conducted even 60 000 more (Święcki 1992). About 20% complications occurred after them were connected with the problem of the total blocking of the lumbar spine (Marciniak 2002). Mostly, implants used in the spinal operations are produced from titan alloy, which is not radiopacity. That means that the medical staff is not able to check the proper implementation in human body or to assess the reason of different complications after operation because of many artefacts which occur due to metal implants. Subsequently, scientists found the material, which is strength, biocompatible and radiodensity with smaller amount of Young modulus what makes the spine connection less rigid. As a consequence of research, scientists and doctors found new material – polyether-ether-ketone, which is commonly used also in different fields of industry like building machines (Turner *et al.* 2002).

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PEEK material is a thermoplastic polymer with halfcrystallic structure. For the first time, it was commercially used in 1980 and then, in 1998, it was applied to the medical services. Nowadays, PEEK material is commonly used in lots of surgical fields, e.g.: spine surgeries, orthopedy.

Implants which are produced with the use of polietheroetheroketon are divided on five groups (Panayotov *et al.* 2016):

– Spine implants;
– Orthopaedic implants like bone implants;
– Stomatology implants;
– Implants used in face or scalp transplantation;
– Heart valves, inner heart pumps.

PEEK material is chemically neutral and high temperature resistance. That is why it can be sterilized, what makes PEEK a very good material for the use of biomedical engineering.

1. Materials and test procedures

Prefabricated plates of PEEK material delivered for the tests were available in two thickness configuration: 12 and 10 mm. The scheme and main dimensions of the PEEK beam for SENB specimen is shown in Figure 1. The same type of prefabricate was delivered for CT specimen with the thickness t = 12 mm).

The results of static tensile test are presented in Table 1. There are shown also the mechanical properties of PEEK material which are provided by the producer of this material.

Fracture toughness tests were performed using CT (Compact Tension) specimen. Specimen size and methods of testing (Fig. 2) were defined according to the guidelines of ASTM D5045-14 standard in order to satisfy the plane strain regime criterion. Following criterion had to be fulfilled (ASTM 2014):

$$B, a, (W-a) > 2.5\left(\frac{K_Q}{\sigma_\gamma}\right)^2, \qquad (1)$$

where: B is the specimen thickness, a is the crack length, W is the specimen width, K_Q is the stress intensity factor value corresponding to the P_Q, and σ_y is the yield stress.

In Figure 3 is shown, the instrumented CT specimen with MTS extensometer mounted on the additional external knifes subjected to the fracture toughness test. Mechanical notch of the specimen was prepared using milling machine. In both cases (CT and SENB)

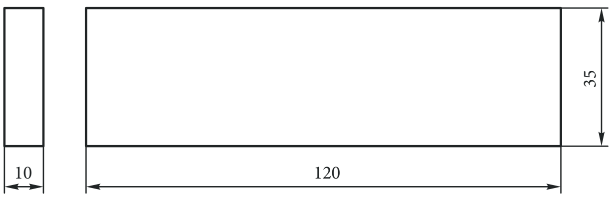

Fig. 1. Shape and dimensions of the PEEK prefabricate (thickness of SENB specimen – 10 mm, CT specimen – 12 mm)

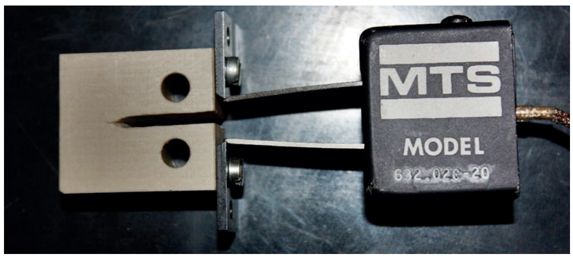

Fig. 3. CT specimen of PEEK material used in the fracture toughness test (thickness: B = 12 mm)

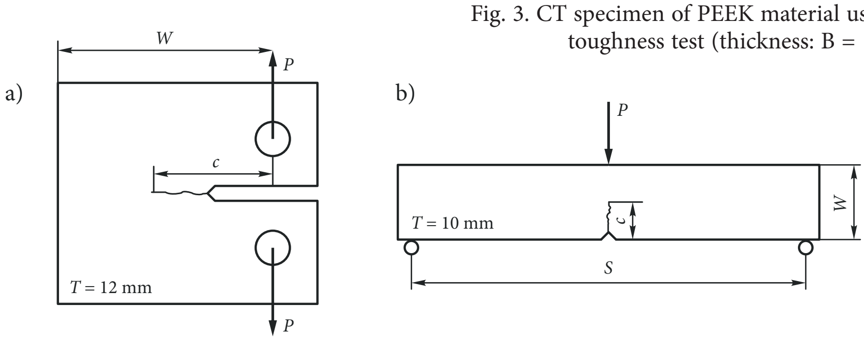

Fig. 2. Specimen configuration for fracture toughness tests: a) CT specimen, W = 30 mm, T = 12 mm; b) SENB specimen, W = 20 mm, T = 10 mm

Table 1. Mechanical properties of investigated PEEK

	Young modulus E [MPa]	Poisson's ratio ν [–]	Yield strength σ_y [MPa]	Tensile strength σ_u [MPa]	Elongation A [%]	Impact test KCV [kJ/m^2]
tested PEEK	4044.0	0.34	116.0	116.0	15.0	4.0

notch was V-shaped, straight through with the height h = 2 mm. Before the proper test, notch was sharpened using surgical blades. During pre-crack preparations, sufficiently sharp crack was generated by sliding a new blade across the notch root. The blades were changed several times in order to maintain a sufficiently sharp crack tip with the valid "pre-crack length".

Fracture toughness tests were conducted using servo hydraulic MTS BIONIX testing machine (load cell ±5 kN). The tests were performed using displacement control method – cross-head speed was kept on the level 3 mm/min. The aim of this research was to register value of the load (*F*), displacement (*u*) and crack opening displacement (COD). The representative Force-COD curve (for specimen CT-01) is presented in Figure 4.

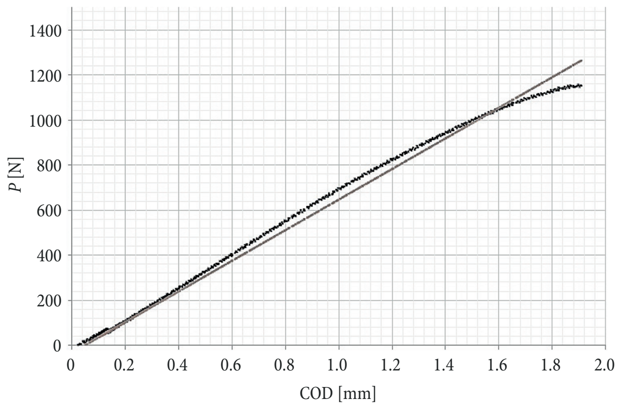

Fig. 4. Representative load-COD curve as a result of fracture toughness test (K_{IC}) with the use of CT specimen

Beyond the load-COD curve, an additional curve is also presented in Figure 4. Tangent of the additional curve is 5% less than angle tangent of the slope of the curve in the beginning of the graph. On this basis, the load P_Q was determined and the criterion could be checked what is necessary in order to find the proper value of K_Q or K_{IC}:

$$\frac{P_{\max}}{P_Q} \leq 1.1. \quad (2)$$

In all cases (three specimens), these criteria were fulfilled. Therefore, it was possible to calculate stress intensity factor for compact specimen according to the following equation (ASTM 2014):

$$K_I = \frac{P_q}{BW^{1/2}} \frac{(2+A_w)\left[0.886+4.64A_w-13.32A_w^2+14.72A_w^2-5.6A_w^4\right]}{(1-A_w)^{3/2}}, \quad (3)$$

where: P_q – value of the load determined by the experiment; A_w – normalised dimension of the crack; W – width of the specimen; B –thickness of the specimen.

For the tested specimens, the value of K_Q were following: 6.63; 6.72; 6.94 MPa√m with the thickness (*B*) equal to: 12.01; 12; 11.96 mm. In order to check if the value of K_{IC} can be the same as K_Q, was necessary to apply the criterion of the plane state of strain (1) for the smallest value of *B, a, W-a*.

Finally, this criterion was fulfilled. The calculated value of K_Q is equivalent to K_{IC} and is equal to 6.76 MPa√m.

Additionally, there were performed tests of the fracture toughness with the use of SENB specimens (the thickness of the material equal to *B* = 10 mm). The crack was prepared in the same way as it was further on the CT specimens. However, in this case was also observed the damage zone (so called process zone) ahead of a crack tip using Digital Image Correlation (DIC) techniques. Shape and dimensions of the specimen used during the tests are shown in the Figure 5. The measurement stand is presented in the Figure 6.

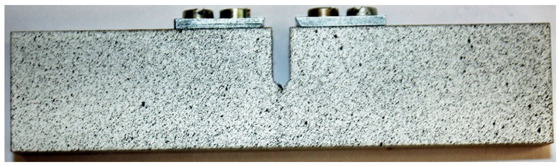

Fig. 5. SENB specimen prepared for the fracture toughness test during the three point bending flexural test

Fig. 6. The machine with the experimental setup for DIC measurements

Before the proper test, the speckle pattern was prepared on the specimen surface. The specimen was painted on the white colour and the black dots were sprayed randomly in order to obtain the correct contrasted speckle pattern for deformation analysis. The photogrammetric idea of 2D-DIC is shown in Figure 7. During the experiment the images focussed on ROI (Region of interest) were captured in order to determine the deformation maps and strain distribution.

The digital image correlation works tracking small square subsets of an unloaded specimen – reference image to the images of the surface after deformation process. The general rule is illustrated in Figure 8 by means of a series of mathematical mapping and cross correlation functions. It is worth to underline the difficulties to distinguish every single pixel on the image and therefore at least 3×3 pixels are needed for one recognizable feature. The ideal subset size should contain at least three clear features but it is often a compromise between resolution and accuracy (Yates *et al.* 2010).

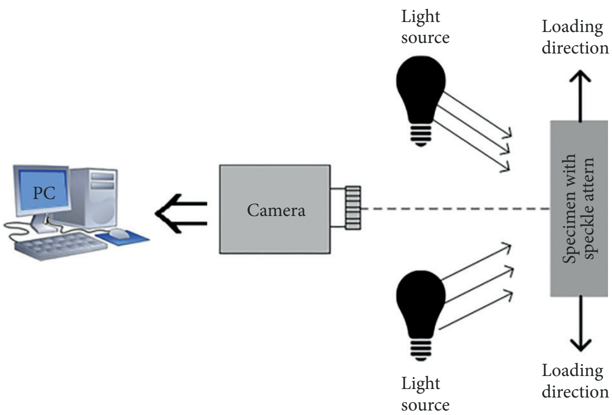

Fig. 7. Measurement scheme of 2D DIC system

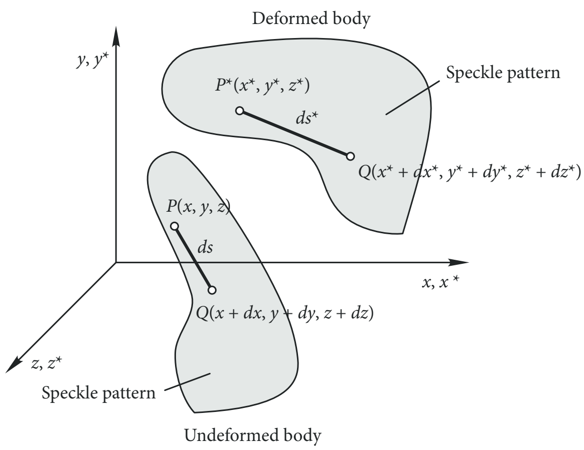

Fig. 8. Line segment PQ before and after deformation

Generally, it can be state that larger subset sizes will increase the accuracy whereas a smaller subset will increase the resolution but realistically the size of a subset is determined by the quality of the image and speckle pattern (Yates *et al.* 2010).

From the mathematical point of view DIC method focuses on the analyse of short segments locations described by distance PQ and P*Q* presented in Figure 8. (Chu *et al.* 1985), (Lagattu *et al.* 2004). Each point is determined in the coordinate system $P(x,y,z)$ and $Q(x+dx,y+dy,z+dz)$. After deformation of the specimen, the position of P* and Q* is following (Chu *et al.* 1985):

$$P^* = \left(x^*, y^*, z^*\right) = \left[x+u(P), y+v(P), z+w(P)\right]; \quad (4)$$

$$\begin{aligned} Q^* &= \left(x^*+dx^*, y^*+dy^*, z^*+dz^*\right) = \\ &\left[x+u(Q)+dx^*, y+v(Q)+dy^*, z+w(Q)+dz^*\right] = \\ &[x+u(P)+u(Q)-u(P)+dx, y+v(P)+v(Q)- \\ &v(P)+dy, z+w(P)+w(Q)-w(P)+dz]; \end{aligned} \quad (5)$$

$$|PQ|^2 = (ds)^2 = dx^2+dy^2+dz^2; \quad (6)$$

$$\left|P^*Q^*\right|^2 = \left(ds^*\right)^2 = dx^{*2}+dy^{*2}+dz^{*2}. \quad (7)$$

Hence:

$$dx^* \cong \left(1+\frac{\partial u}{\partial x}\right)dx + \frac{\partial u}{\partial y}dy + \frac{\partial u}{\partial z}dz; \quad (8)$$

$$dy^* \cong \left(1+\frac{\partial v}{\partial y}\right)dy + \frac{\partial v}{\partial x}dx + \frac{\partial v}{\partial z}dz; \quad (9)$$

$$dz^* \cong \left(1+\frac{\partial w}{\partial z}\right)dz + \frac{\partial w}{\partial y}dy + \frac{\partial w}{\partial x}dx, \quad (10)$$

where u, v, w denote the displacement vectors. According to above, it is possible to calculate the deformation and strain tensors for two-dimensional measurements with the use of DIC (Lagattu *et al.* 2004); (Chu *et al.* 1985). For this purpose, the GOM-Correlate® system was used.

The SENB specimen was used for fracture tests. The sharp pre-crack was introduced using surgical blades. The displacement control mode was applied during the experiment with the crosshead speed 6 mm/s. During the experiment following signals were registered: load (F), displacement (u), crack opening displacement (COD). Additionally, the images focused on the ROI were captured (sampling frequency – 5 Hz) using monochromatic PointGrey FL3-U3-13Y3M-C camera (pixel size: 4.8×4.8 μm) with lenses. During experiment the stable cold light LED source was applied.

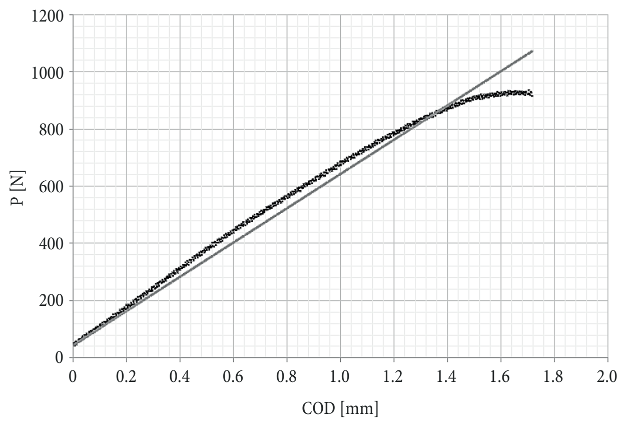

Fig. 9. Load-COD curve as a result of fracture toughness test (K_{IC}) with the use of SENB specimen

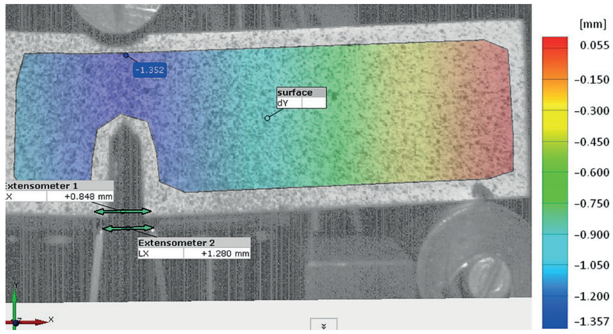

Fig. 10. Map of displacement u_{yy} of the specimen corresponding to the force value equal to P_Q = 850 N

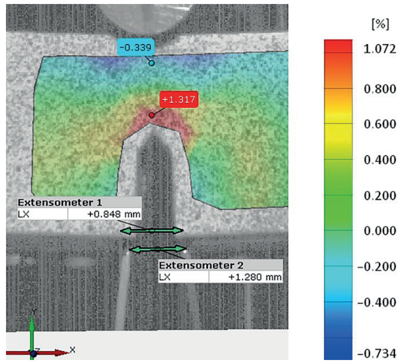

Fig. 11. Map of strain ε_{xx} of the specimen corresponding to the force value equal to P_Q = 850 N

The Force-COD load curve is presented in Figure 9. According to obtained data, the criterion (2) was fulfilled. Then it was possible to calculate the value of K_Q for specimen which was used during the three point bending flexural test in accordance with:

$$K_Q = \frac{P_q S}{BW^{3/2}} \frac{3\left(A_w\right)^{1/2}\left[1.99 - A_w\left(1-A_w\right)\left(2.15-3.93A_w+2.7A_w^2\right)\right]}{2\left(1+2A_w\right)\left(1-A_w\right)^{3/2}}. \quad (11)$$

In Equation (11) S determines the distance between supports in three point bending flexural test. Final result of this test was the value of K_Q = 8.17 MPa$\sqrt{\text{m}}$. Unfortunately, if we take into consideration the thickness of the specimen (B = 10 mm), the criterion (1) is not fulfilled.

Post-process analysis in GOM-Correlate environment allows to determine the displacement maps, deflections and strain distribution. The exemplary results corresponding to the P_Q force values are shown in Figure 10 and Figure 11.

Beyond the small plastic zone, the fracture surface is largely brittle with isolated regions of river markings. It is worth noting that the region suffered intense whitening due to plastic deformation before crack. The fracture surface of the sample CT after K_{IC} test is shown in shown in Figures 12–15. The surface of the crack is divided on two typical zones: small tensile zone with the elements of tensile mechanism cracks and much bigger zone of fast; and dynamic crack expansion with dominating influence of fragile fracture zone. Crack surface in the initial expansion was perpendicular to the main direction of the loading is shown in Figure 13. A noticeable is crazing mechanism with the numerous of river pattern associated with brittle nature of fracture. On the further part of the material crack surface is characterized (Fig. 14) by the rapid change of the crack surface orientation and its bifurcation. This effect is related to the structural construction of PEEK material (Kurtz 2012). The magnified region of river pattern is shown in Figure 15 – typically for fast, brittle fracture.

Fig. 12. Macroscopic view of fracture surface – CT specimen

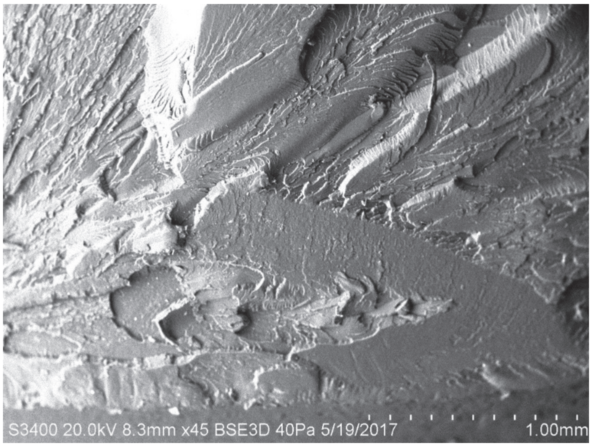

Fig. 13. Microscopic view on the initial crack surface (CT sample) after the fracture toughness test, SEM image in BSE technique, direction of the crack propagation: from the bottom to the top

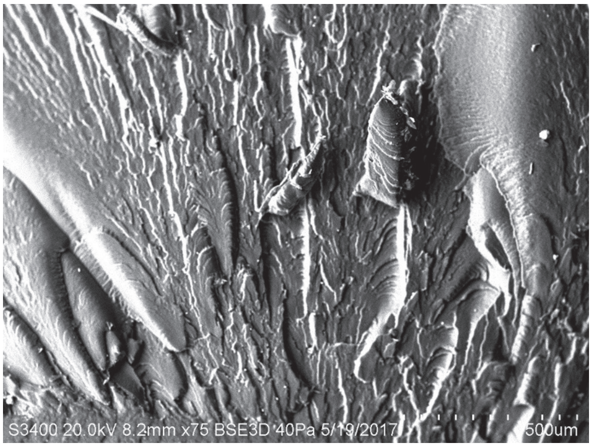

Fig. 14. Microscopic view on the fast fracture region (CT sample) after the fracture toughness test, SEM image in BSE technique, direction of the crack propagation: from the bottom to the top

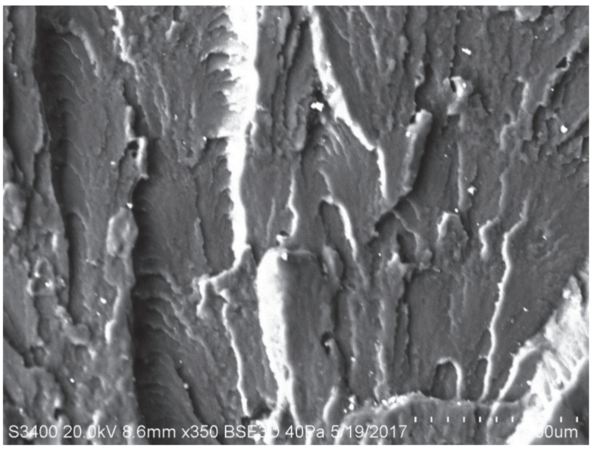

Fig. 15. Magnified area from the Figure 14 – a microscopic view on the fast fracture region characterized by river pattern area (CT sample) after the fracture toughness test, SEM image in BSE technique, direction of the crack propagation: from the bottom to the top

Summary and conclusions

The aim of this work was to present the results of studies on PEEK material with the use of fracture toughness test and digital image correlation. In order to conduct the tests, there were used two types of sample: SENB and CT.

Averaging all the results of the tests, critical value of stress intensity factor K_{Ic} is about 6.76 MPa$\sqrt{}$m for CT specimens. Due to lower thickness – for SENB specimen critical value of stress intensity factor (as it was expected) is higher K_Q = 8.07 MPa$\sqrt{}$m.

It should be state, that in comparison with other polymeric materials subjected to biomedical application, PEEK material presents good fracture resistance with their biocompatibility.

It is predicted that in the future the development of PEEK materials will be associated with the 3D printing methods in the field of tissue engineering. PEEK is already being used to produce end-use implantable medical devices, but many different problems to apply these devices in the clinic are persisting. The 3D printed PEEK medical devices will not possess the same biomechanical and surface properties like the CAD/CAM produced ones (Panayotov *et al.* 2016), Therefore the obtained results are reference results for further investigation (also with multiaxial stress state consideration).

Acknowledgements

The experimental works have been supported in part by Department of Mechanics, Materials Science and Engineering, Wroclaw University of Science and Technology (no. 0401/0029/17).

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