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## The safety at work in experiments of polymers and composites

Bezpieczeństwo pracy w eksperymentach z polimerami  
i kompozytami

### Abstract

The paper deals with safety at work during special tests for polymers – polymer fibres and long-fibre composites at on selected laboratory test machines and apparatus. The operators have to be very careful during the test and measure process. We described TGA/DSC analyzer for polymer testing and laboratory line for plasma surface modification and safety at work with this low-temperature plasma reactor. Next, the paper described safety at work at laboratory for the production of elastomeric and composites samples which consists from torque rheometer, homogenizing machine, vulcanization hydraulic press and pneumatic hollow die punch. Test samples of elastomers and composites with polymer fibres are the product of this laboratory. These test samples are then tested on universal testing machine with hybrid temperature-humidity chamber.

**Key words:** *safety of work, test, composite, polymer*

### Streszczenie

Artykuł dotyczy bezpieczeństwa pracy podczas specjalnych testów polimerów - włókien polimerowych i kompozytów o długich włóknach w wybranych laboratoryjnych maszynach i urządzeniach testowych. Operatorzy muszą być bardzo ostrożni podczas procesu testowania i pomiaru. Opisaliśmy analizator TGA/DSC do testowania polimerów oraz linię laboratoryjną do modyfikacji powierzchni plazmy i bezpieczeństwa pracy w tym niskotemperaturowym reaktorze plazmowym. Następnie w artykule opisano bezpieczeństwo w pracy w laboratorium przy produkcji próbek elastomeru i kompozytów, które składa się z reometru momentu obrotowego, maszyny homogenizującej, prasy hydraulicznej wulkanizacji i pneumatycznego wykrojnika. Próbkki testowe elastomerów i kompozytów z włóknami polimerowymi są produktem tego laboratorium. Te próbkki testowe są następnie testowane na

uniwersalnej maszynie testującej z hybrydową komorą temperatura-wilgotność.

**Słowa kluczowe:** *bezpieczeństwo pracy, test, kompozyt, polimer*

## Introduction

The paper deals with safety at work during special tests for polymers – polymer fibres and long-fibre composites at on selected laboratory test machines and apparatus. The safety and warning information on injuries are provided on these devices. High temperatures are applied to the test equipment, and some have moving parts. Therefore, the operators have to be very careful during testing, measurement and production of test samples too.

## 1. Safety at work at TGA/DSC analyzer to find the crystallinity temperature of polymers

The DSC measurements are carried out using a TGA/DSC 2 HT/1100 STA-Re System (Mettler Toledo, Schwerzenbach, Switzerland), see Figure 1.

The measurement procedure is: the test samples such as polymer fibres are prepared by cutting to very small stripes of approximately  $20 \pm 2$  mg weight and pressed into aluminum crucibles of volume 70  $\mu$ l. Subsequently, they are heated from 50 to 250°C at a heating rate of 10 °C/min in nitrogen atmosphere under the flow rate of 20 ml min<sup>-1</sup>. Thus, a melting endotherm of sample with melting temperature ( $T_m$ ) and melting enthalpy ( $\Delta H_m$ ) are obtained. Then the samples are held at 250°C for 5 min to remove the thermal history of the polymer fibre preparation. The sample are then cooled to temperature 50°C at a cooling rate of 10 °C/min and the crystallization exotherm with the crystallization temperature ( $T_c$ ) and crystallization enthalpy ( $\Delta H_c$ ) were obtained.

The melting enthalpy ( $\Delta H_m$ ) are also used for the calculation of degree of crystallinity  $X_c$  (%) of the samples according following equation [1]:

$$X_c = \frac{\Delta H_m}{\Delta H_m^+} \times 100 \quad (1)$$

During measurements, there is a hot part on the analyzer with pictogram (see Figure 1, left) because temperature range is from 30 to 600 °C. The operator must be cautious during measurements and when changing the test samples.



Fig. 1. TGA/DSC testing analyzer with detail of hot part (left)  
Rys. 1. Analizator testowy TGA / DSC ze szczegółami części gorącej (po lewej)

Source: own research.

Źródło: opracowanie własne.

## 2. Safety at work at plasma reactor for surface modification of polymers

Plasma reactor KPR 200 (Figure 2) using DCSBD (Diffuse Coplanar Surface Barrier Discharge) plasma systems with flat and curved electrode with active plasma area 200 x 100 mm is used for surface modification of surface of polymers too[2]. A unique feature of the plasma source based on DCSBD is a possibility of generating homogenous plasma under atmospheric pressure with virtually any working gas composition without usage of expensive inert gases. Extremely high power density of plasma up to  $100 \text{ W/cm}^3$  allows short plasma exposure times and thus high speed processing. This allow plasma reactor KPR 20 produced by Research Institute for Man-Made Fibers (Svit/Slovak Republic).

Plasma can be used for continuous double-sided surface modification of thin flat materials (polymers, textiles, metals, glass) and composites (foils with thickness of  $50 \mu\text{m}$ -0.5 mm, thin flexible polymeric flat plates with thickness of 0.5-1 mm and thin textile materials as well as cords with diameter of 0.1-0.5 mm). The biggest advantages of using low-temperature plasma in comparison with other “physical” methods of activation of polymer fibre surface are low costs, high speed of the process and efficiency up to the depth of about 10 nm. Plasma action on nanometric level on polymer fibre surface allows to create quite a new class of innovative materials and their use. It is environmentally friendly process because no chemicals are used.



*Fig. 2. Plasma laboratory equipment*

*Source: own research.*

*Rys. 2. Sprzęt laboratoryjny*

*Źródło: opracowanie własne.*

Contact with the human body and the plasma electrode system is not dangerous, but unless the contact occurs, it is not uncomfortable. During the plasma treatment the ozone is formed, but ozone is exhausted. The devices are always tailor-made for specific customer applications and are necessary to focus on work safety. Our plasma reactor has several drawbacks as unprotected rotating parts of the equipment. There is possibility of electrostatic charges and possibility of improper use of electrical wiring. The on/off (total stop) button is only on the control panel. There is possibility of burning during operation of the device. It has a high sound level.

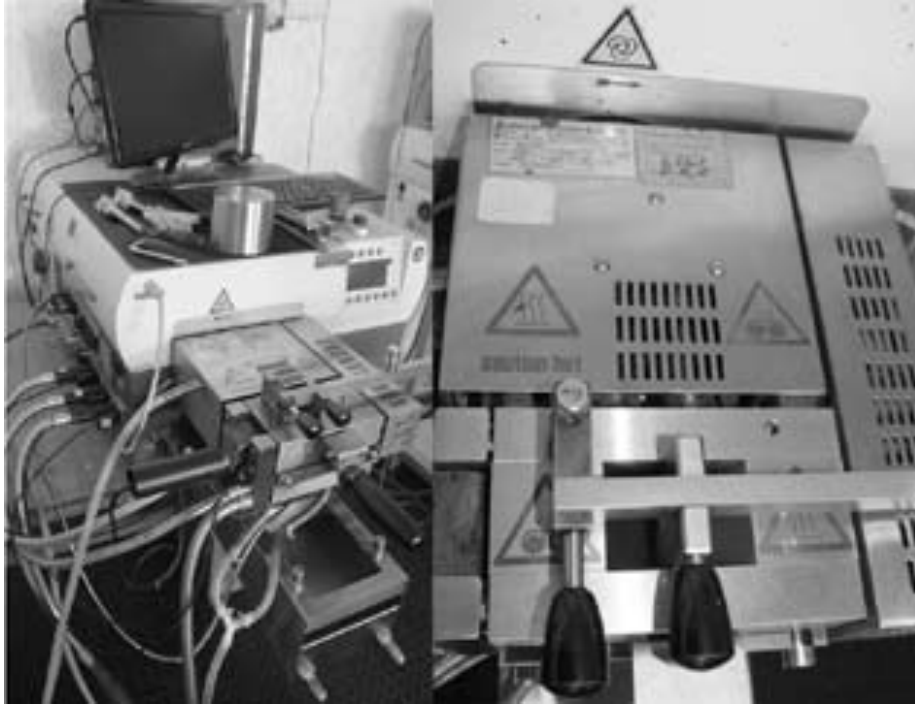
### **3. Safety at work at laboratory for the production of elastomer and composite samples**

The laboratory consists from:

- torque rheometer for mixing and extruding elastomers,
- homogenization machine,
- vulcanization hydraulic press,
- pneumatic hollow die punch.

The torque rheometer Brabender Plastograph® EC plus (Figure 3) is used for simulation of elastomer processing and manufacturing procedures under the laboratory conditions – heating, blending, mixing, reactive mixing, kneading. There is a very hot chamber (working temperature can be up to 500°C) with

rotation parts with revs up to  $150 \text{ min}^{-1}$ . During machine running the operator must be very careful. Risk of burns.

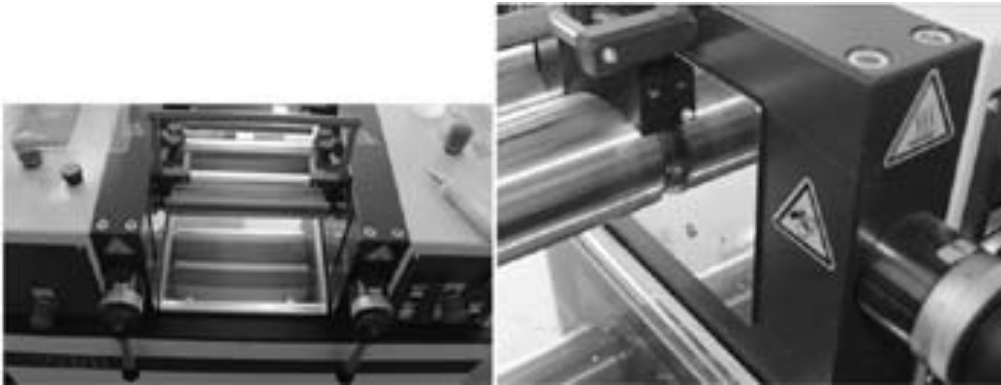


*Rys. 3. Reometr momentu obrotowego ze szczegółami komory gorącej z piktogramami  
Źródło: opracowanie własne.*

*Fig. 3. Torque rheometer with detail of hot chamber with pictograms  
Source: own research.*

As next step, there is homogenization process of elastomer. The homogenizing machine from company Vogt at Figure 4 is used. The temperature is changed during homogenization process (maximum temperature is  $150 \text{ }^\circ\text{C}$ ). There are very hot rotating parts – two heated rollers. In addition, hair and clothing of operator may be pulled in, see pictograms on machine.

As next step, there is vulcanization process on vulcanization hydraulics press LabEcon 600, see Figure 5. The product is vulcanizing plate[3] with defined thickness. The press is fully automatic and the operator is protected by a safety cover. The maximum temperature is  $300^\circ\text{C}$  but standard temperature for vulcanization process of elastomer is from  $150$  to  $180^\circ\text{C}$ . In addition, emergency buttons are located.



*Fig. 4. Homogenization machine with detail of rotating heated rollers and hot parts (right)*

*Source: own research*

*Rys. 4. Maszyna do homogenizacji ze szczegółami obracających się podgrzewanych rolek i gorących części (po prawej)*

*Źródło: opracowanie własne.*



*Fig. 5. Full automatically vulcanization hydraulics press*

*Source: own research.*

*Rys. 5. Prasa hydrauliczna z pełną automatyczną wulkanizacją*

*Źródło: opracowanie własne.*

After press, the test samples with standard shapes according to ISO 37[4] (see example at Figure 6 left) from the vulcanizing plate are made on pneumatic hollow die punch Inston, see Figure 6 right. There is a risk of injury to the fingers, so the control buttons for run of device are located on both sides of the device and must be pressed by the operator with both hands at the same time. Thus,

the moving part cannot come into contact with the operator's fingers.

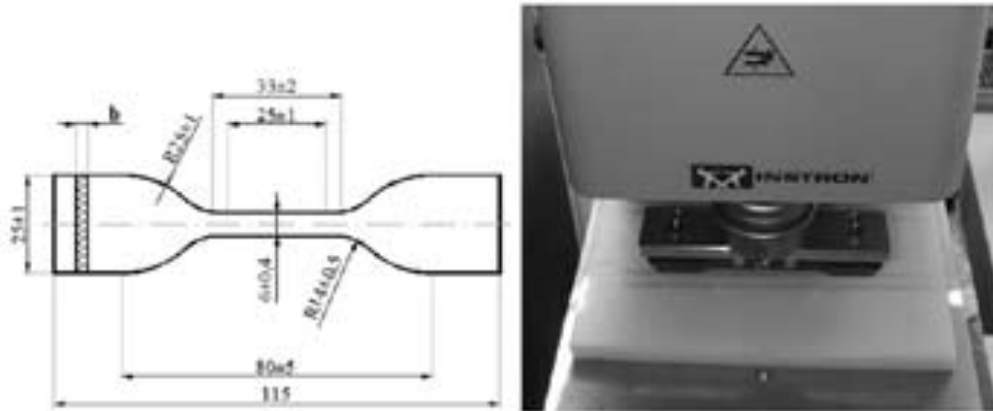


Fig. 6. Pneumatic device for the production of test samples with shape of samples (left)

Source: own research.

Rys. 6. Pneumatyczne urządzenie do produkcji próbek testowych (po lewej)

Źródło: opracowanie własne.

For prepare polymers from granulates, the torque rheometer Brabender Plastograph® EC plus with measuring extruder with six temperature zones is used, Figure 7. The extruder is very hot during mixture and extrude process.



Fig. 7. The measuring extruder of torque rheometer for production of polymers

Source: own research.

Rys. 7. Wytłaczarka pomiarowa reometru momentu obrotowego do produkcji polimerów

Źródło: opracowanie własne.

#### 4. Safety at work during tensile tests of polymer fibres and composite samples

The Autograph AG-X plus 5kN – Shimadzu Japanese testing machine with hybrid temperature-humidity chamber (Figure 8) and special jaws (see Figure 8

c) is used for the tests of polymer fibres[5]. The chamber allows to perform the tests from -60 to 180°C, but it is important to point out that from 20 to 80°C, it is possible to change the humidity from 30 to 95%.

The operator has to be really careful because there is a risk of finger injuries during closing of the compressed-air (pneumatically) jaws. Also, the operator risks of hand burning (risk of burning or risk of frostbite) during change of samples because high or low temperature is in the during tests and after tests. The start/off button as emergency button is at control panel of chamber and emergency buttons is at testing machine.



Fig. 8. Universal testing machine (a) with hybrid chamber (b) and detail of special jaw for fibres (c)

Source: own research.

Rys. 8. Uniwersalna maszyna testująca (a) z komorą hybrydową (b)  
i szczegółami specjalnej szczęki do włókien (c)

Źródło: opracowanie własne.



## Conclusion

Each testing device/machine must be secured with emergency buttons. Pictograms must be provided on each device and the operator must be properly trained.

Nevertheless, the operator must be cautious because there are rotating / moving parts during measurement or prepare of test samples on devices/testing machines such as work on homogenization machine and can be high temperatures used for experiments such as measurement on TGA/DMA analyzer. Therefore, students are not allowed to work on these devices.

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## Bibliography

- [1] Jambrich M., *Štruktúra a vlastnosti vlákien*. 1987, Bratislava: SVŠT. 542 p., in Slovak.
- [2] Krmelová V., R. Janík and I. Kopal. Operation of DCSBD plasma reactor in laboratory conditions. *Zeszyty Naukowe Wyższej Szkoły Zarządzania Ochroną Pracy w Katowicach*. 2018, 14(1), pp. 95-103. ISSN 1895-3794.
- [3] Krmela J., *Tire Casings and Their Material Characteristics for Computational Modeling*, scientific monograph, Czestochowa, Poland: Oficyna Wydawnicza Stowarzyszenia Menadżerów Jakości i Produkcji (Printing House the Managers of Quality and Production Association), 2017, 109 p., ISBN 978-83-63978-62-4.
- [4] ISO 37. Rubber, vulcanized or thermoplastic – Determination of tensile stress-strain properties.
- [5] Krmela J., V. Krmelová and A. Artyukhov. Safety at work during cyclic loading tests of composites, tests of tires and printing on 3D printer. In: *Księga dobrych praktyk BHP*. Katowice: Wyższa Szkoła Zarządzania Ochroną Pracy w Katowicach, 2018, pp. 173-186. ISBN 978-83-61378-52-5.