# Experimental ceramic proppants characterization in the process of shale gas extraction

Charakterystyka eksperymentalnych propantów ceramicznych służących w procesie pozyskiwania gazu z łupków

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The presented paper relates to the application of new experimental proppants in the process of shale gas extraction. The tested proppants were produced through mechanical granulation methods. Ceramic proppants were sintered at 1550 °C. The characterization of investigated proppants was carried out using Scanning Electron Microscope (SEM) and Light Microscope (LM). Properties of ceramic proppants such as: bulk density, crash tests, moisture, solubility in acids, sphericity factor, turbidity were studied using a range of techniques. The results show that new experimental proppants meet the industrial requirements and need some modification for future application.

#### KEYWORDS: ceramic proppants, mechanical granulation, shale gas

Artykuł przedstawia wyniki badań eksperymentalnych propantów ceramicznych z przeznaczeniem do wydobywania gazu z łupków. Otrzymane propanty zostały poddane spiekaniu w temperaturze 1550 °C. Otrzymane w ten sposób próbki poddano badaniom mikrostruktury SEM, gęstości nasypowej, wytrzymałości mechanicznej, wilgotności, odporności na kwasy, współczynnika kulistości oraz mętności, z użyciem przeznaczonych do tych badań technik. Stwierdzono, iż nowo uzyskane propanty spełniają założenia przemysłowe, jednak wymagają modyfikacji w celu zastosowania ich do wydobywania gazu z formacji łupkowych.

SŁOWA KLUCZOWE: propanty ceramiczne, granulowanie mechaniczne, gaz łupkowy

The growing energy demand forces to discover and develop new technology and beds for energy production in the word. In past, when unconventional deposits of shale gas where discovered for the first time, its extraction from shale rock had a marginal share in the natural gas processing  $[1\div3]$ .

For economic reasons and the lack of techniques for exploitation of the new beds, shale gas plays a small role in energy industry. Today, through a better understanding of mining technology and beds geological identification, new energy sources are very desirable in the energy market.

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For various reasons the extraction of shale gas from Polish beds is very difficult. One of very important causes is the location of beds. Polish shale gas is located very deeply in the ground. Depth reaches up to 3 km. Therefore acquisition of shall gas is very expensive. The light weight ceramic proppant can provide a solution for this problem. This specific materials can be applied in hydraulic fracturing method. They are characterized by spherical shape and are increasingly used for extraction of shale gas from beds in US by injecting them to boreholes. These granulates pumped with liquid into the deposit cause destruction of rock with shale gas. Due to this process, proppants must exhibit suitable physico-mechanical properties  $[1, 4\div6]$ .

## Methodology

This research was conducted utilizing new ceramic proppant sintered in 1550 °C and based on Polish raw materials. To characterize microstructure, Hitachi SU-70 with 10 kV voltage and YAGBSE detector was used. Evaluation of particle shape was measured using MicroMeter 1.04 software, and ceramic proppants pictures taken on Nikon DS-F12. Turbidity measurements were made using a Turbi-Direct\_4a, wherein the beam of light is perpendicular to the detector. The volume of measured sample was 20 ml. To study the solubility in acids, HCI:HF acids with the ratio 12:3 were used. Bulk density and crash tests were performed according to Polish Standard PN-EN ISO 13503-2. Moisture analysis was conducted in RadWag moisture analyzer.

#### **Results and Discussion**

Fig. 1 shows SEM and LM images of investigated ceramic proppant. It was fabricated using Polish raw materials in granulation method. Images present large proppant particle with the shape similar to the sphere. Many different particle sizes of investigated proppant were observed in LM image (Fig. 1*c*). EDS results of characterized proppant showed, that the main components are aluminium and silicon oxides (Fig. 1*b*).

To measure the average equivalent diameter (0.89 mm), one of the Nikon DS-F12 photos (Fig. 2) was used. The sphericity of studied proppant is 0.84.

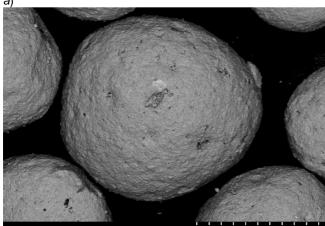
Basic properties of studied proppant were characterized in Tab. I. Investigated proppant exhibits adequate properties which are within the range of values accepted by the industry.

Fig. 3 and Tab. II summarize the crash tests results. Tests speed of the piston was 1 mm/min. Holding matrix time was 2 min. For the first sample, the pressure of 50 MPa

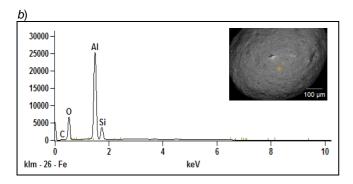
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was used, for the second one – 75 MPa and for the third one - 100 MPa. For these pressures, samples exhibit the strain values of 105 kN, 142 kN, 181 kN, respectively.

a)



10.0kV 20.1mm x120 YAGBSE



C)



Fig. 1. SEM and LM images of investigated proppants: *a*) SEM images, *b*) EDS results, *c*) LM images

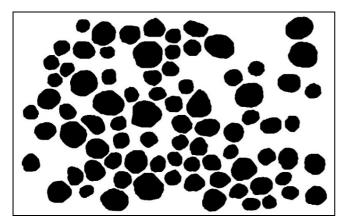
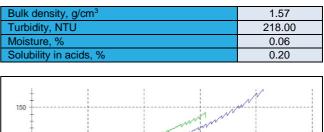


Fig. 2. LM image of investigated proppants used to measure equivalent diameter and sphericity factor

#### **TABLE I. Basic properties of investigated proppants**



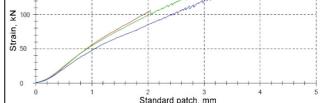


Fig. 3. Crash test results for investigated proppants

#### **TABLE II. Crash tests parameters**

Sample No.	Strain, kN	Standard path, mm
1)	105	2.05
2)	142	3.12
3)	181	4.17

## Summary

Experimental ceramic proppants obtained by mechanical granulation method are characterized by appropriate parameters, i.e. low bulk density, solubility in acid and a relatively high value of sphericity -0.84. These granules provide the basis for application in the hydraulic fracturing process.

However, obtained proppants did not exhibit very high strength. The amounts of destroyed granules were: for 7500 psi – 5.5%, for 10000 psi – 21% and for 15000 psi – over 30%. These values are unacceptable by the industry. Additionally, researched proppants revealed too high value of turbidity, therefore they need further refinement for future application.

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