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Investigations of the Rock-Polymer Composites as a Gasket for HPHT Apparatus

Abstract
In the HPHT apparatus, pressure is generated using gaskets, which are made from soft minerals, such as lithographic stone, catlinite or pyrophyllite. Polish metamorphic rocks (metamorphic schist, amphibolite) were used as solid pressure transmitting media. The rock-resin composites were pressed into pellets of 15 mm in diameter. The sintered materials were thermally treated at 120°C. SEM observations have showed that the rock-resin composite structure was compact, and that the binding phase (resin) was uniformly distributed between the rock's grains. Mechanical tests confirmed the high mechanical properties of the prepared composite materials. The composites investigated are very good raw materials for producing ceramic gaskets to generate high pressure in the HPHT apparatus.

Key words: *HPHT apparatus, rocks, lithographic stone, catlinite, pyrophillite, amphibolite, metamorphic schist, SEM, WAXS, mechanical investigations.*

Introduction

High-temperature apparatus

High pressure is a very effective tool for research into discovering new phenomena and exploring new materials.

Two types of ultra-high pressure, high-temperature apparatus are in use at the present time. The first of these, the 'Belt,' uses conical 'pistons' thrust into opposite ends of a properly-shaped and -gasketed chamber, that is, surrounded by a toroidal belt' of supporting rings.[1, 2]. The second device is the 'Tetrahedral Anvil Apparatus.' [3-5]. This device is geometrically the simplest of a series of related devices which make use of polygonal anvils that enclose regular polyhedra when fitted together about a common point. Pressure is generated by forcing the anvils against a polyhedral cell. Three subjects related to these and other high-pressure apparatus will be considered in the present work: (1) gaskets for obtaining relative motion of anvils or other pressure-generating components, (2) solid pressure transmitting media, and (3) high-tonnage hydraulic rams.

Gaskets

Three important functions are performed by the gasket:

- 'yielding' to the thrust placed on it by a moving anvil or similarly functioning apparatus component. Yielding can occur through simple compression of the gasket material, by flow, or by a combination of compression and flow;
- 'confining' (i.e. not yielding to the thrust of) the material being compressed by the advancing anvil;
- 'supporting'. Inside the inner edge of the gasket, the surfaces of the high-pressure apparatus components are subjected to the maximum possible pressure generated within the chamber. Pressure gradient patterns can be varied by the choice of gasket materials, and by changing the cross sectional profile of the gasket from the inner to the outer edge.

Ideally, a solid pressure transmitting medium should meet the following requirements:

- transmit pressure hydrostatically.
- have very low compressibility.
- have very low thermal conductivity.
- have very low electrical conductivity.

Figure 1. *Outside part from catlinite.*

Figure 2. *Central part from catlinite.*

__Table 1.__ Basic properties of the rock used for gasket preparation.

Characteristic feature	Kind of rock	
	Amphibolite	Metamorphic Schist
Type	Metamorphic	Metamorphic
Origin	Ogorzelec, 'Ogorzelec' mine	Krobica, 'Jerzy' mine, Orlowice
Structure	From fine to coarse-grained schistose, massive	From fine to medium-grained, schistose, lamellar
Main component	Amphiboles, plagioclase	Quartz, muscovite, biotite, paragonite
Colour	Black, dark grey, dark greenish	Yellowish, brown, grey
Density, g/cm^3	2.95	2.91
Compression strength, Mpa	202.77	129.57
Use in the present time	Highway engineering	Paint filler, carrier for plant pesticides

- have a very high melting point, which should increase with increasing pressure.
- be chemically inert.
- be thermally stable.

Of course, it is impossible to find a solid pressure transmitting medium wherein all these requirements are fully and simultaneously met. Consequently, a compromise must be effected. At the present time, gaskets in traditional Kvostantsev cells are made from soft minerals such as lithographic stone or catlinite, Figures 1,2, (materials based on $CaCO_3$) or pyrophillite. Sometimes the gasket is made in dual form, with a central part from catlinite and a toroidal ring from pyrophillite [6]. Each of these materials has its advantages and disadvantages.

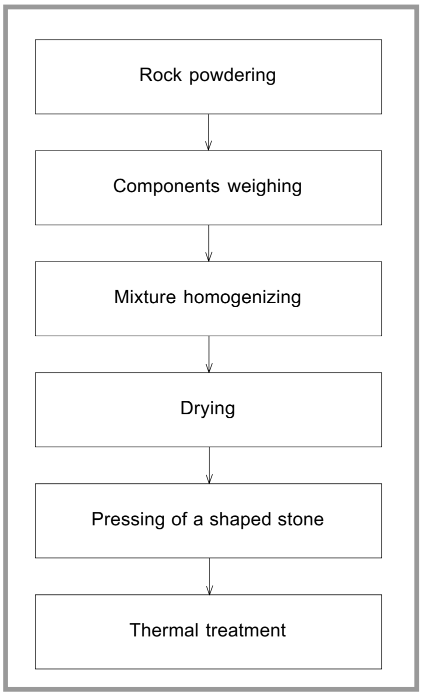

***Figure 3.** Technological steps of ceramic gasket preparation.*

Polish metamorphic rocks

Most frequently, metamorphic rocks belong to the group of magmatic or sedimentary rocks transformed in the earth by the action of high pressure and temperature. Due to these transformations, their structure and mechanical properties change. These are usually crystalline rocks which usually show random or linear (oriented in one direction) textures.

Amphibolite

Amphibolite is the name given to a rock consisting mainly of amphibole (hornblende), the use of the term being restricted, however, to metamorphic rocks. Hornblende is usually dark green but may be nearly black in the hand specimen; on the microscopic slide it is commonly a green of various shades, but may be brown, blue or nearly colourless. Amphibolite from the Ogorzelec region is composed mainly of plagioclase and hornblende. Quartz and mica occur with it simultaneously.

Sometimes the rock also contains calcite, ironstone, apatite, magnetite, hematite, etc. Amphibolite is commonly applied in highway engineering.

Metamorphic Schist

Claystone and mudstone rocks are the starting materials for metamorphic schist. Schist from Krobica is composed of muscovite and water-muscovite, which are accompanied by grenads and quartz as well as chlorite. In some parts of schist, small amounts of iron and tin minerals appear. The rock exhibits very low endurance to abrasion and crushing.

Comparison of the selected materials is presented in Table 1.

Experiment

Polish metamorphic rocks (schist, amphibolite) were used as solid-pressure transmitting media. In the experiments, the rocks and resin in a 10:1 weight ratio were mechanically mixed in ethyl alcohol and then pressed into pellets of 15-mm diameter using a pressure of 11MPa. The sintered material were thermally treated at 120°C for 1 hour. The technological steps for preparing ceramic gaskets for use in high-pressure sintering are presented in Figure 3.

Results and discussion

Studies of compression strength

Mechanical tests were performed in a conventional Instron test machine, fitted

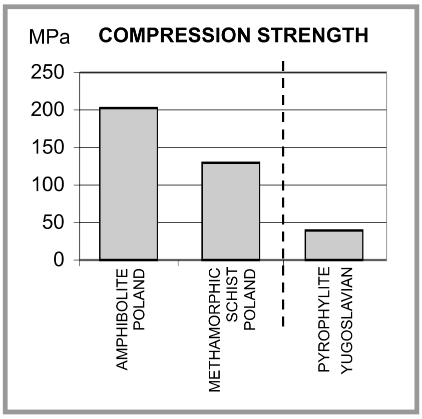

***Figure 4.** Comparison of the compression strength of rocks from Poland and the former Yugoslavia.*

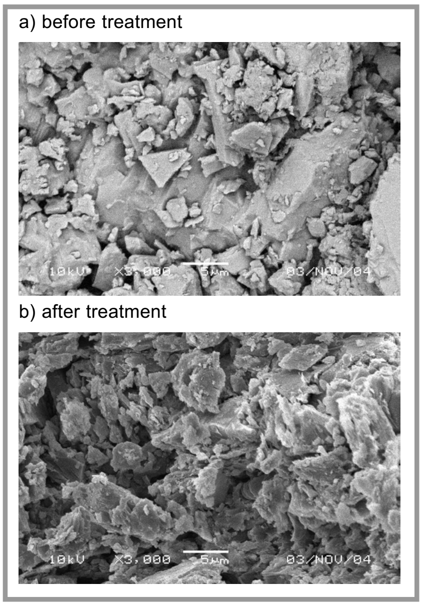

***Figure 5.** SEM microstructure of Amphibolite-resin composite, thermally treated at 120^{o}C for 1 hour.*

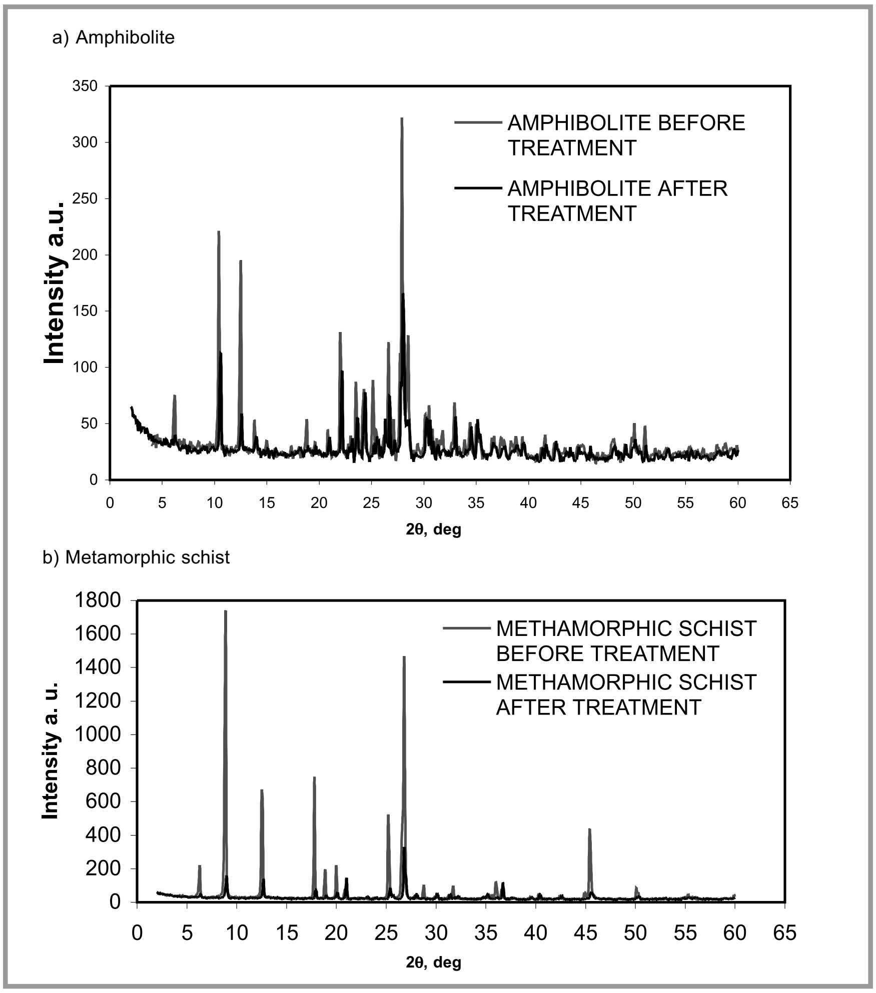

Figure 6. *XRD analysis of gasket materials before and after treatment, (thermally treated at 120°C for 1 hour).*

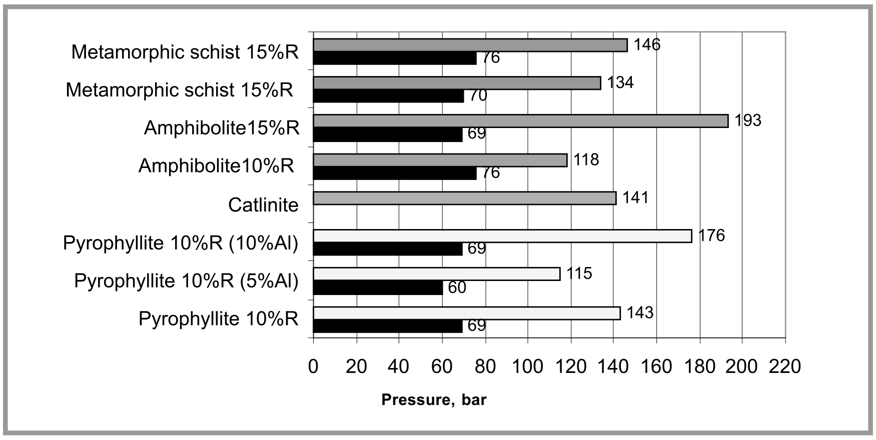

Figure 7. *Testing pressure gaskets.*

with compression plates. They confirmed the high mechanical properties of the composite materials prepared (*Figure 4)*

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Morphological (SEM) studies

SEM observation showed that the rock-resin composite structure is compact, and the binding phase (resin) is uniformly distributed between the rock's grains (Figure 5). This analysis confirmed the correctness of the technological process used for the preparation of ceramic gaskets.

Wide-angle x-ray diffraction studies of rocks and ceramic gaskets

Wide-angle X-ray diffraction (WAXS) *Figure* studies were carried out (Figure 6) using a Seifert URD-6 diffractometer equipped with a scintillation counter as a detector. Ni-filtered CuK_α radiation was applied. Diffraction patterns were measured in the 2θ range from 2° to 60°, with the step of 0.1°, and a counting time of 5s. This technique was used to study the content of phases in the rock-resin samples.

3.4. Testing pressure gaskets

Gaskets made of rock-resin composites were tested in a Bridgman-type HPHT apparatus.

The pressure values at low temperature in the above gaskets were calibrated by the pressure dependence of bismuth electrical resistance.

The rock-resin gaskets investigated in the present work are very good for generating high pressure in the HPHT apparatus. (Figure 7).

Conclusions

The structure of Polish rock-resin composites is compact, and the binding phase is uniformly distributed between their grains.

The composites investigated are characterised by very good mechanical properties.

Polish rocks such as amphibolite and metamorphic schist are very good materials for the production of ceramic gaskets to generate high pressure in an HPHT apparatus.

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Acknowledgment

This study is based on the work sponsored by 6th Framework European Programme, project NAMAMET (STREP, No 001470)

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❑ ***Received 08.12.2004 Reviewed 10.02.2005***