Gas Analysis of Magnetorheological Elastomer During Vacuum Degassing

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Abstract. Research conducted in a vacuum requires high accuracy, but multiple vibrational and shock disturbing effects adversely affect the results. To increase the accuracy, it is possible to install a vibration isolation system in a vacuum chamber. One of the promising areas of active vibration isolation is the use of intelligent materials in them, including magnetorheological elastomer (MRE). Since most studies need to be carried out in a high and ultra-high vacuum environment, it is necessary to know the degree of influence of the MRE gas release on the quality of the operation environment. Thus, the aim of the work was to analyze the spectrum of released gases during degassing of the polymer (MRE) in vacuum at room and elevated temperatures. It was found that increased gas evolution from the studied polymer sample with increasing temperature does not allow to achieve high vacuum ($1.7 \cdot 10^{-29}$). We also identified that gases with the highest partial pressures are part of the atmospheric air; there are other gases not analyzed that significantly affect the total pressure (have a pressure of the order of $10^{-29} - 10^{-28}$).

1. Introduction
Among intellectual (“smart”) materials, magnetorheological elastomers (MRE) are one of the promising groups of composites. They represent a class of materials with controlled rheological properties i.e. their viscosity, elasticity and plasticity vary depending on the external magnetic field applied to them [1].

The first studies of magnetorheological elastomer were conducted in the 1950s by Jacob Ryabinov in the USA. The composition of MRE is not standardized; various combinations of polymer components are possible. The main elements of MRE is a polymer matrix and filler particles. Experiments on the manufacture of magnetorheological elastomer of various compositions are presented in [2,3,4]. The options of possible filler particles (e.g., FeNdB, Fecarb), as well as their concentrations are shown in these works. The analysis of the compositions allowed us to determine the main components for the manufactured samples. The most commonly used polymers are based on carbonyl iron particles. Particle material has an elongated shape, which contributes to the greatest deformation and rapid magnetization reversal, the optimal concentration of filler particles is 30%.

A study of the mechanical properties of a magnetorheological elastomer is given in [5,6,7,8]. Experimental stands with the possibility of studying deformation under the action of a directed magnetic
Examples of polymer loading are given to study the possibility of deformation under static and cyclic loads. The magnetic, static and dynamic properties of experimental samples are studied. MRE is used in systems of vibrational protection, in the works [9,10,11,12] the most common constructions of polymer application are given: sensors; converters; automotive bushings and engine mounts; shock protection; self-adjusting vibration absorber.

At the Department of MT11 (BMSTU), an analogue of modern active vibration isolation systems based on the MRE is being developed [13,14]. The modes of operation of this platform and the change in the characteristics of the damper based on the MRE are considered - stability, time constant, positioning error, transmission coefficient of vibration displacement amplitude [15,16,17]. This platform can be applied in a vacuum to protect the objects against vibration in order to improve the accuracy of research and improve the technological capabilities of equipment. To date, the gas evolution of the polymer, including during its degassing, has not been sufficiently studied. The gas evolution of a small sample of a magnetorheological elastomer has previously been considered; experiments showed the possibility of using the polymer in a vacuum [18,19,20].

Thus, the aim of this work was to study the gas evolution of magnetorheological elastomer samples, the sizes of which differ significantly from previously studied. It was necessary to prepare the vacuum pumping station for research, to develop the design of the test chamber, to carry out two test cycles with different warming temperatures, accompanied by heating the chamber, holding at a given temperature and cooling.

2. Methods
MRE gas evolution experiments were carried out at a high-vacuum pumping station with a bell chamber. To conduct the experiment, a chamber was designed with the possibility of placing specimens for tension and compression in it simultaneously, as well as with a volumetric pumping volume [21]. Partial gas pressures were recorded using an Extor 200 quadrupole mass spectrometer.

Preliminarily, on the basis of the laboratory of SSC JSC “GNIIHTEOS”, tensile specimens were manufactured in accordance with GOST R 56785-2015 (Polymer composites. Methods of tensile testing of flat specimens) and compression in accordance with GOST 33519-2015 (Polymer composites. Test methods for compression about normal, high or low temperatures) [22].

3. Preparation of Equipment for Conducting Degassing Experiments
Tests to study the gas evolution of the magnetorelastic elastomer were carried out on the basis of the laboratory of NPK Pluton JSC, at a high-vacuum pumping station with a bell chamber (Fig. 1). Pumping is carried out in two stages: fore-vacuum pumping - rotary vane two-stage monoblock pump 2NVR-5DM, high vacuum pumping – magnetic discharge vacuum diode cooled pump - NDMO-01-1. During the operation of the post, the pressure was displayed on the screen of the control unit according to the pressure gauges (wide-range vacuum gauge - SS - 10; thermistor - PMT - 6 - 3M - 1). An Extorr 200 quadrupole mass spectrometer connected to a computer recorded the total pressure in the pumped volume and the partial pressures of the gases specified for the study.
Figure 1. General view of the high vacuum pumping station (1-mount of a tubulation; 2 – bell-shaped vacuum chamber with heaters; 3 – control unit)

For testing, samples were selected for tensile and compression, the placement of which in the tubulation on this installation is impossible. For experiments, as well as for the order of NPK Pluton JSC, a vacuum chamber was designed. Since the installation is a production one, the design change was to be minimal and reversible, so it was necessary to design the camera with fastening on a copper tubulation.

To implement the connection of a copper tubulation with a steel chamber, a circuit similar to the connection with a copper seal of the ConFlat type was proposed (CF) (fig. 2). The connection was established in accordance with GOST 26526 - 85 on vacuum equipment. The upper flange of this connection has CF type teeth, which, when connected to the lower flange, deform the copper plate pre-connected to the copper tubulation, thereby ensuring sealing of this connection.

Figure 2. Coupling a copper tubulation with a steel flange (1 – upper flange; 2 – lower flange; 3 – copper plug; 4 – copper tubulation)

To place the samples in the chamber, two hooks were provided that fixed the samples in a predetermined position during pumping (fig. 3). After placing the samples and sealing the upper flange of the chamber (CF copper connection), the design was placed in the plug socket of the installation (fig. 4).
4. Degassing of Magnetorheological Elastomer

One tensile specimen and one compression specimen were placed in the chamber simultaneously (fig. 3). After that, the chamber was pumped out, when the pressure on the magnetic discharge pump of about $10^{-7}$ Torr was reached, the heating process began. Heating continued for 15 minutes, after which exposure was carried out at a given temperature for 30 minutes and uncontrolled cooling of the chamber in air [23,24].

Two consecutive experiments were carried out with heating up to $100^\circ$C and up to $200^\circ$C for different pairs of samples (one for compression and one for tension). A mass spectrometer recorded the partial pressures of the 13 most common gases with atomic masses: 1 u (H); 2 u (H₂); 12 u (C); 14 u (N); 15 u (CH₃); 16 u (O + CH₄); 17 u (OH); 18 u (H₂O); 24 u (C₂); 28 u (N₂ + CO); 32 u (O₂); 40 u (Ar); 44 u (CO₂).

5. The results

According to the pressure gauges, graphs of the dependence of the total pressure on time in the chamber during pumping were constructed (fig. 5 and fig. 6). The graphs show that the pressure at the beginning of the process (at the start of heating) at different temperatures is similar. Upon reaching the holding temperature, a sharp increase in pressure is observed, which indicates the intensification of gas evolution from pores and from the surface of MRE samples. At the beginning of exposure, the pressure increase is greatest, after 20 minutes the gas evolution rate drops, in Fig. 6 the pressure drop begins. Comparing the pressure during degassing at different temperatures, it is noticeable that at a heating temperature of $200^\circ$C the total pressure in the system reaches about $10^{-3}$ Torr. -vacuum at this pressure can no longer be considered high.

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**Figure 3.** Placement of samples in the chamber (1 – holders; 2 – samples)

**Figure 4.** Placement of the camera at the pumping station (1 – chamber; 2 – tubulation; 3 – tubulation holder)

**Figure 5.** Chamber pressure during warming up to $100^\circ$C and shutter speed

**Figure 6.** The pressure in the chamber during heating to $200^\circ$C and shutter speed
To analyze the gases that have the greatest influence on the total pressure, graphs of the dependence of pressure on time for each of the 13 studied gases were constructed, all the pressures are of order $10^{-6} \ldots 10^{-7}$ Torr (fig. 7 and fig. 8). According to the partial pressure graphs, similar to the total graphs, it is clear that before the start of the degassing process, the pressures practically do not change. When heated up to 100°C, after the start of the process of degassing, the gas pressure decreases, there are gas peaks indicating gas release from the pores of the MRE. When heated up to 200°C, a large number of gas peaks do not allow one to judge the degree of pressure reduction by the end of the degassing process. This behavior indicates significant gases in the pores of the samples and other impurities, which are most intensely released with increasing temperature.

![Figure 7. Graphs of partial pressures of gases during heating to 100 °C and exposure](image1)

![Figure 8. Graphs of partial pressures of gases during heating to 200 °C and exposure](image2)

The sum of the partial pressures of the studied gases at a certain point in time differs from the total pressure, which indicates the presence of gases unaccounted for in the study, which have the greatest influence on the studies [25].

After carrying out the experiments and extracting samples from the experimental chamber, oily spots were found on its internal surfaces, and the samples became more dense. Excessive impurities that negatively affected the mass spectrometer readings were probably removed from the material during the degassing process.

6. Conclusion

The designed chamber with tubulation pumping allows to carry out tests at a laboratory pumping station without changing its design. CF copper connection allows high vacuum testing.

Based on the data obtained, it is impossible to unambiguously determine the possibility of using the polymer in a vacuum. With increasing temperature, the flow of gas from the MRE increases, the total pressure in the chamber does not reach the high vacuum level.

Most of the evolved gases are part of atmospheric air and can be removed from the material with longer degassing. In the composition of the evolved gases there are specific ones that have the greatest influence on the degree of vacuum. In the future, it is planned to carry out similar experiments with an extended range of studied gases and to analyze the components removed during pumping.

To conduct further experiments, recommendations will be made on the composition of the polymer, in order to reduce impurity components. It is supposed to conduct experiments with longer exposure and heating to higher temperatures.
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