Practical methods for the experimental determination of the mechanical characteristics of cement stone under compression

D Lobanov, E Strungar, Y Kurbatov, E Zubova, V Wildemann, and G Kashevarova

1Perm National Research Polytechnic University, Russia

E-mail: wildemann@pstu.ru

Abstract. In this work, we propose practical methods for the experimental study of the mechanical characteristics and staging of damage accumulation of cement stone based on the joint use of modern testing equipment and a non-contact optical video system for analyzing displacement and deformation fields, as well as a system for recording acoustic emission signals. In the framework of these practical methods, compression tests of cement stone samples of various shapes and geometrical sizes were carried out. The diagrams of deformation and application of loading have been obtained, the mechanical characteristics have been determined, and the staging of damage accumulation in a cement stone has been analyzed. We have received a conclusion about the optimal shape of samples of cement stone, which can be used to determine the prism strength, elastic modulus and Poisson's ratio using the proposed practical methods.

1. Introduction

Today, concrete is one of the most important structural materials in modern construction. It is known that the main indicators of the structural strength of concrete, which reflect its operational characteristics, vary ambiguously with a change in structural parameters. According to [1], such parameters include the mechanical properties of cement stone, aggregate characteristics, types of additives, etc. Thus, studies of strength should be based on establishing the dependence of the effective properties of cement composites on the geometric and physical characteristics of the phase components which contain in them.

According to V.V. Fleckenstein and several other researchers [2], the main mechanism of the destruction of cement stone is its cracking caused by circumferential and tension stresses. To create a shock-resistant cement stone, the authors propose to be guided not only by compressive strength, but also by tensile and shear strength. At the same time, in order to reduce circumferential stresses and improve the deformation properties of cement stone, it is necessary to increase the Poisson's ratio and reduce the elastic modulus of this material. Today, concrete is one of the most important structural materials in modern construction. It is known that the main indicators of the structural strength of concrete, which reflect its operational characteristics, vary ambiguously with a change in structural parameters. According to [3], such parameters include the mechanical properties of cement stone, aggregate characteristics, types of additives, etc. Thus, studies of strength should be based on establishing the dependence of the effective properties of cement composites on the geometric and physical characteristics of the phase components which contain in them.

In work [4], to analyze the mechanical properties of cement stone, such parameters as elastic modulus, creep strength, and tensile strength were chosen. Moreover, to estimate the elastic modulus,
the $\sigma$-$\varepsilon$ curves in the linear section were approximated by the dependence: $\sigma=\text{A}+\text{G} \cdot \varepsilon$, where $\sigma$ - applied stress; $\varepsilon$ - deformation; $\text{A}$ - a constant. $\text{G}$ it characterizes the intensity of hardening and allows researchers to evaluate the modulus of elasticity of the samples. Thus, the need for determining the elastic response of cement stone becomes apparent. The indicated parameters can be used as initial data in mathematical modeling of the process of destruction of a concrete structure. So, in work [5], the authors proposed a structural-simulation model of a cement-sand composite, which includes pores, cement stone, sand and the contact zone between them. As a result of the numerical calculation of this model, integral elastic characteristics of the material were obtained, which later could be used as initial data for modeling a higher scale level in order to optimize the composition of the concrete. In this case, the elastic characteristics of the main structural components of the model were taken on the basis of reports in literature. To date, several existing GOSTs and other regulatory documents are devoted to determining the strength and elastic characteristics of concrete material as a whole - for example [6,7] and several others. However, only one current standard regulates the experimental determination of the strength characteristics of cement stone - this is [8] “Cements. Methods for determining the tensile strength in bending and compression”. According to the instructions of this GOST, as samples, bending test beams with sizes 160x40x40 should be used, which are prepared in the corresponding demountable molds. To test the beam samples for bending, you can use devices of any design that support the average rate of rise of the test load on the sample in the range of $0.05 \pm 0.01 \text{ kN /s}$ [0.12±0.02] MPa/s in terms of unit reduced cross-sectional area of the test beam.

To determine the compressive strength of samples, presses of any design with a maximum load of up to 500 kN can be used to ensure loading of the sample in pure compression mode. The specified GOST clearly identifies the rules for the manufacture, storage and testing of samples in order to determine the tensile strength of cement stone for bending and compression. In the future, the tensile strength can be used to assign the appropriate grade of cement. Unfortunately, this GOST does not regulate the determination of the elastic characteristics of cement stone, as a result of which there is no clear understanding of the requirements for the shape, size of the samples, as well as the algorithm for testing if it is necessary to determine the elastic modulus and Poisson's ratio of the cement stone.

A promising area of experimental mechanics is the joint simultaneous use of test and complex measuring systems. To study defects, cracks, peelings arising in structural materials, such promising methods are used as ultrasonic flaw detection, X-ray tomography, infrared thermography, acoustic emission (AE), optical and fiber-optic methods for measuring deformations, etc. In works [1-10] the possibility of joint usage of acoustic emission, infrared thermal imaging, non-contact optical system for analyzing fields of deformation and an optical fiber system with gauges for deformation measuring based on Bragg gratings for various types of tests is shown.

In the framework of this work, the goal was set - to develop a methodology for the experimental determination of the mechanical characteristics of cement stone during compression based on the use of modern testing and measuring systems, based on the recommendations of current standards in the field of construction. Experimental studies were carried out using the large-scale research facilities «Complex of testing and diagnostic equipment for studying properties of structural and functional materials under complex thermomechanical loading» PNRPU modernized with funds by the Ministry of Science and Higher Education of the Russian Federation, Unique project identifier RFMEFI61920X0017.

2. Material and Samples Preparation

To experimentally determine the mechanical characteristics of cement stone, the following sample options were made (Figure 1): cubes (70x70x70), prism test beams (160x40x40 и 140x70x70), and cylinders (diameter 30 mm, length – 60 mm). Prism test beams and cylinders were made to determine the prism strength and elastic characteristics of cement stone, cubes - to determine the tensile strength in accordance with [8]. When choosing the shape and size of the samples, emphasis was placed on the instructions of the relevant GOSTs for testing concrete samples.
In accordance with [9], all samples were made of cement mortar with W/C = 0.40 and a consistency characterized by a cone flow diameter of 106 .. 115 mm. Demountable molds for test beam samples were made of metal that met the conditions of their operation and ensured rigidity of molds and dimensional stability of the samples. The longitudinal and transverse walls of the mold, when fastened, were tightly adjacent to each other and to the pallet, preventing water leakage out of the mold during the manufacture of samples. The limits of allowable wear of the walls of the molds were not more than 0.2 mm in width and height.

Immediately before the manufacture of the samples, the inner surface of the walls of the molds and the palette was lubricated with machine oil. Joints of the outer walls with each other and with a pallet of mold were smeared with a thin layer of dense grease. To seal the mortar, the mold of the test beams with the nozzle was fixed in the center of the vibrating platform, tightly pressing it to the plate. The mold in its whole height was filled with 1 cm of mortar and the vibration plate was turned on. During the first 2 minutes of vibration, all mold cavities were uniformly filled with a mortar solution in small portions. After 3 minutes from the start of vibration, the vibratory plate was turned off. The mold was removed from the vibrating plate and the excess mortar solution was removed with a knife located at a small angle to the laying surface. After that, the samples were marked and placed in storage in a chamber for normal hardening (Figure 2, a), providing a relative humidity of at least 90%.

After 24 hours of storage in the chamber, the samples were carefully dismounted and placed in a bath of drinking water in a horizontal position so that they did not come into contact with each other (Figure 2, b). At the same time, water covered the samples by at least 2 cm. Water was replaced every 14 days of storage. After 28 days of storage in the bath, the samples were extracted from water, wiped, measured and tested within 30 minutes after extraction.
3. Methods and Equipment

The experimental studies were carried out on a universal electromechanical system Instron 5989 with a maximum load of 600 kN, using the Vic-3d digital optical system for analyzing deformation and displacement fields (Correlated Solutions) and the AMSY-6 acoustic emission signal recording system (Vallen-Systeme GmbH). The mathematical apparatus of a non-contact optical system for analyzing the fields of displacements and deformations is based on the method of digital image correlation (DIC). This is a non-contact optical method for recording the fields of displacements and deformations on the surface of an object. All information is contained in the structure (pixel distribution) on the surface of the test object with varying degrees of brightness of the black-and-white image. Due to the fact that the DIC method is based on the idea of measuring the deformation of a material by tracking the distortion of the pattern of random points on the surface, the surface of the sample should have a contrasting finely dispersed color, which is applied to the sample before testing.

For the experiment, it is necessary to configure the test equipment, the test setup is shown in Figure 3. Two sets of digital cameras are mounted on a rigid frame so as to exclude movement of one camera relative to the other, and the frame is attached to a tripod to fix the camera at the required height. As a light source, fluorescent lamps are used, the sharpness of the cameras is adjusted. Video recording of samples during the test is carried out using the Vic-Snap software package, which allows the researchers to set the parameters of the image-taking process: frame rate, recording duration, as well as adjust the synchronization of cameras and the controller of the test setup (synchronization ensures the simultaneous operation of cameras and the controller of the testing machine).

Information is recorded automatically on the computer’s hard drive for subsequent processing of digital photos in the Vic-3D software package. The software is based on the Digital Image Correlation method. Before starting the test, it is necessary to calibrate the cameras. After shooting, it is necessary to process digital photographs of the displacement and deformation fields in the Vic-3D software package using the Digital Image Correlation method.

![Figure 3. Instron 5989 test system before testing and the test setup in conjunction with a non-contact optical video system: 1—test machine, 2—sample installed in the grips, 3—test system controller, 4—PC from which the machine is controlled, 5—synchronization block, 6—PC from which the video system is controlled, 7—cameras installed on tripod, 8—backlight system.](image)

The video system was shot using two Q-400 digital black and white cameras with a resolution of up to 4 megapixels, the shooting speed was 15 frames per second. The software of the Vic-3D video system provides for the use of different correlation criteria for the mathematical assessment of the conformity
of digital images. In the work there is a usage of the criterion of the normalized sum of squared difference (NSSD). In the correlation processing of digital images, displacement vector calculations are not carried out at each individual image point (pixel), but by discretizing the study area into small local subareas or subsets of \( n \times n \) pixels.

The choice of the size of the subset and the step is carried out in accordance with the conditions of the recording made, with the results of the calibration of the stereo system, as well as depending on the geometric parameters of the object of study and the structural features of the material of the sample.

A significant influence on the construction of displacement and deformation fields is exerted by the parameters of correlation image processing. From the data obtained during correlation processing, it was found that a subset of size 27 \( \times \) 27 for the cube, 31 \( \times \) 31 for the cylinder and 35 \( \times \) 35 pixels for the prism test beam is optimal for the image of these structures. The choice of step determines the detail of the formation of the displacement and deformation fields and is equal to 5 pixels in all cases. During post-processing with the Vic-3D system, the components of the deformations were calculated using the finite strain tensor in the Lagrange representation

\[
\varepsilon_{ij} = \frac{1}{2} (u_{ij} + u_{ji} + u_{ik}u_{kj}).
\]

When preparing the surface of the sample, a contrasting finely dispersed color was applied to the surface using white and black opaque spray paint. For this, a white matte base was first applied to the surface of the samples, after which a combination of black dots was applied.

Also, during the mechanical testing of the samples, continuous monitoring was carried out using the acoustic emission method. The method of acoustic emission is based on the registration and analysis of elastic waves arising in the process of deformation during application of loading onto the material. To record acoustic emission signals during application of loading onto the samples, we used a broadband piezoelectric sensor (frequency range 450–1150 kHz), which was fastened with high-vacuum silicone grease to the side surface of the sample (Figure 4) and a preamplifier (gain ratio of 34 dB). The sampling frequency of the data is 10 MHz; the threshold value for recording acoustic emission signals was 34 dB.

![Figure 4](image-url)

Sample images showing the system for recording acoustic emission signals AMSY-6 with a PC and specialized software (a), and an acoustic emission sensor on the test sample (b).

Samples in the shape of cubes were tested in order to determine the tensile strength of the material with a constant speed of movement of the moving grip 2 mm/min. Samples in the shape of a prism test beam (160x40x40 и 140x70x70) and cylinders (diameter 30 mm, length 60 mm) were tested to determine the elastic characteristics by the step regime of loading application with time delays (GOST 24452-80).
4. Results of Testing and Discussions

According to the method described above, tests were conducted on the static compression of samples of cement stone of various shapes. Typical diagrams in the “stress-displacement U” axes are presented in Figure 5, where the “U” axis is the displacement of the moving grip.

![Figure 5. Typical diagrams of loading application during compression of cement stone samples of various shapes.](image)

When determining deformations, GOST 24452-80 recommends the use of strain gauges or conductive strain gauges glued to the surface of concrete. Vic 3D software allows the researchers to simulate strain gauges using the “virtual extensometer” tool (Figure 6). The principle of its action is similar to a strain gauge and consists in tracking the mutual displacement between two points of the surface of the samples in accordance with the applied force. The main advantages of using a “virtual extensometer” are non-contact registration of deformations, which eliminates the mechanical impact on the surface of the sample; the possibility of using several extensometers on one sample, which in turn increases the accuracy of the recorded data.

![Figure 6. “Virtual extensometers” in the longitudinal and transverse directions attached on the surface of the sample.](image)
As an example, Figure 7 shows inhomogeneous fields of longitudinal deformations corresponding to points on the graph (Figure 8) at different load levels for the cube sample. The results obtained illustrate the moment of nucleation and development of cracks on the surface of the sample, which, as they are gradually being loaded, lead to complete destruction.

Based on the data obtained, it is possible to judge at what point in time and at what load, as a result of primary destruction, a crack appeared on the surface of the sample and how it propagated.

A similar pattern of field distribution was observed for prism test beams. Figure 9 shows at what level of load, cracks formed on the surface of the sample. As loading is being applied, the formation of single bands of longitudinal deformations in the working area appears which correspond to the crack propagation site on the surface. With further application of loading, the number of longitudinal deformation bands over the entire surface increases.
Figure 9. Inhomogeneous longitudinal deformation fields for a sample in the shape of a prism test beam.

In all cases, there was a violation of the integrity of the contrast finely dispersed coating, which led to the impossibility of further formation of the deformation fields.

Determination of the elastic modulus and Poisson's ratio of a cement stone was carried out by gradual loading with the waiting periods (steps) of the samples to destruction. During application of loading onto the samples, their deformations were measured within the third stage (GOST 24452-80). For the possibility of comparing the results and for greater clarity, the obtained deformation diagrams for each type of sample were reduced to one type (Figure 10). Each graph was deduced from zero and the values were multiplied by (-1). The test results are shown in Table 1.

The video system has an advantage over strain gauges and is more preferable to use, since it allows the researchers to see the whole picture of the distribution of deformations on the surface of the sample, as well as to fix the appearance and propagation of defects - cracks at all stages of application of loading.

During the analysis of acoustic emission signals obtained in the process of mechanical tests for uniaxial compression of cement stone cubic samples, the dependences of the acoustic emission parameters on time were prepared. Figure 11 (a) shows a diagram of acoustic emission hits distribution. Figure 11 (b) shows the distribution of the energy of acoustic emission signals. Figure 11 (c) shows a diagram of the distribution of peak amplitudes of acoustic emission signals. All three graphs are compared with the graph of application of loading for the sample.
Figure 10. The given diagram of the 3rd section (stages of application of loading) for samples in the shape of prisms and a cylinder.

Figure 11. Distribution diagrams of AE hits (a), distribution of the energy parameter of the signals (b), amplitude distribution, combined with the graph of application of loading.
Table 1. The results of compression tests of samples of cement stone of various shapes.

| №  | Sample shape | Elastic modulus, GPa | Poisson’s ratio |
|----|--------------|---------------------|----------------|
| 1  | cylinder     | 29.0                | 0.22           |
| 2  | prism 40x40  | 22.5                | 0.30           |
| 3  | prism 70x70  | 19.5                | 0.19           |

By analyzing the graph of the dependence of AE hits (Figure 11, a), we can assume the rate of damage accumulation and destruction of the cement stone sample. So, as the load increases to the maximum value, the number of AE signals is recorded evenly, but leaps can be noted at $t = 28$ s, $t = 50$ s, $t = 90$ s. The maximum load level on the graph is accompanied by the largest number of registered AE hits. Let’s consider the distribution diagram of the energy (Figure 11, b). It can be noted that each peak in the values of this parameter is associated with the occurrence of cracks in the sample volume. It is noted that these peaks are not accompanied by drops in load values, except for the latter, at $t = 98$ s, at which the sample undergoes a complete break. The largest leaps in energy values were recorded at time $t = 78$ s and $t = 98$ s. These points are marked in Figure 8 (points 4 and 5) and demonstrate the moment of occurrence and germination of a crack on the surface of the sample. Analyzing the distribution diagram of peak amplitudes (Figure 11, c), it can be noted that as the load increases, signal amplitudes also increase.

5. Conclusion

Thus, a compression test method for cement stone samples was developed and tested with the joint use of the Instron 5989 universal electromechanical testing system (600 kN), the Vic3d non-contact optical video deformation fields’ analysis system and the acoustic emission signal analysis system. The diagrams of deformation and loading application of samples of cement stone under compression are prepared. The totality of the systems used allows researchers to determine the mechanical characteristics on samples of any geometry, as well as to track the appearance and development of defects at any stage of loading application, which was shown in the work. The obtained test results allow us to conclude that samples in the shape of prism test beams of 160x40x40 size are optimal for studying the elastic characteristics of cement stone in the framework of this technique.

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