Time dependence of mechanical properties of specimens made from "grey gypsum"

P. Padevět, P. Tesárek, T. Plachý

***Abstract*—** The paper present monitoring of mechanical properties of grey calcined gypsum. The samples of dimension 40 x 40 x 160 mm were made from this material and put in laboratory conditions (relatively humidity 50 % and temperature 20 °C). Material properties, especially Young's Modulus, strength and creep were measured in different time instants during hardening of gypsum samples. At end of paper, the time evaluation of grey calcined gypsum mechanical properties is presented and discussed.

***Keywords*—** Calcined gypsum, mechanical properties, destructive methods, creep of gypsum, shrinkage.

I. INTRODUCTION

Hydration is basic process how made from gypsum (calcium sulphate hemihydrate $CaSO_4 \cdot ½H_2O$) to hardened gypsum (calcium sulphate dihydrate $CaSO_4 \cdot 2H_2O$). Hydration, it is typical effect for hydraulic binders. During this process, hydration heat is generated and a volume increases – expansion. Hydration is set off after mixing water with gypsum.

The process of gypsum hydration and setting relies on multiple factors:

- the temperature during preparing of the gypsum paste,
- the water-gypsum ratio,
- the method of gypsum mixing,
- the mixing intensity and time,
- the fineness of grinding,
- the purity of gypsum binder [1].

The water-gypsum ratio has an influence on the basic physical characteristics of hardened gypsum, such as its volume density, total open porosity and other related characteristics like its moisture, mechanical, thermal and sound insulation properties. The theoretical water-gypsum ratio necessary for the hydration of calcium sulphate hemihydrate into calcium sulphate dihydrate is 0.187. Additional water, in a so-called over-stoicheiometric quantity, is necessary for the processing of the hardening gypsum paste. Depending on the value of the water-gypsum ratio, the gypsum is made from a gypsum paste by pressing, vibrating or casting [2].

The purity of gypsum binder corresponds with a relationship between different phases of calcium sulphate-water system; it means a proportion between anhydrite, hemihydrate and dihydrate of calcium suplphate and impurities which contains every gypsum binder [3].

The process of solid structure evolution relates with hardening of gypsum paste. As basic mechanical properties for characterization of gypsum properties are usually used compressive and bending strength, other mechanical gypsum properties, as Young's Modulus, are tested less often [4]. The strength characteristics correspond mainly with physical properties of hardened gypsum as total open porosity, arrangement of gypsum crystals, and type of used gypsum binder [5]. On the other hand, properties of gypsum depend on conditions where the hardened gypsum is placed. Temperature and moisture (relative humidity but especially a liquid water content) prejudice mechanical properties of gypsum. In a first phase of gypsum hydration, time dependence is observed.

Hurmanic and Roggendorf [6] made comparisons between natural gypsum and flue gas desupfurization gypsum. They also compared their mechanical and physical properties: hardness by Vickers, abrasive power, abrasive effect, compressive strength, the size and distribution of pores. These results showed that the artificially produced material had better characteristics than the natural material. More research projects of this type were carried out with the main objective of proving the suitability of artificially produced gypsum, or hardened gypsum respectively. Mechanical properties (compressive strength, tensile bending strength, Young's modulus, Poisson's ratio) are known from the following publications: Klein and Ruffer [7], Ghozh et al., [9]. Verbeek and du Plessis [4] measured the tensile bending strength and volume density of phosphogypsum with various gypsum-cement ratios. Tazawa [10] presents the tensile bending strength, compressive strength and the modulus of elasticity for three types of α- and β-gypsum. Acker [11] compares different methods of determining the mechanical properties

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(tensile bending strength and compressive strength) on gypsum wallboard. Singh and Garg [12] measured compressive strength in relation to pH values of gypsum. In the area of gypsum applied by dentists, Li et at. carry on experiments with cold pressing of gypsum (300 MPa for a time of 10 minutes and successive placement of samples in a water environment for one hour). The samples treated in this way reached strength values three times higher than the reference samples.

From presented results is visible clear that the mechanical characteristics (above all strength) of gypsum after its hydration rely on the above-mentioned conditions applied during the development of its own solid structure, but also on the successive placement of the unit (for example, the difference between a placement in a water environment and in the air etc.

The strength values of hardened gypsum significantly depend on the water-gypsum ratio, which commonly takes the value for pouring in the interval of 0.6 to 0.8.

Current standards as Czech standard ČSN 72 23 01 Gypsum binders – Classification, general technical specifications and test methods (1979) specify tested compressive strength after two hours (after gypsum was added into water) [6].

One of the important properties of hardened gypsum is a fact, that it is possible take out a gypsum samples from the mould twenty minutes after mixing the gypsum with water. After this time, the gypsum sample (with standardized normal consistence and without admixtures) is self-supporting; it is able to be manipulated and loaded.

After two hours (standard time), the compressive strength of the gypsum samples is approximately 1/5 of maximal compressive strength, which is developed after 28, respectively 14, days after mixing. Time 28 days is standardized for current building materials as plaster, cement or concrete. The laboratory conditions with relative humidity 50 % and temperature 20 °C are standard. In these conditions, gypsum samples are placed [6]. From conditions of a sample deposit, it is clear that the weight of the samples will decline in time. Question is how moisture affects the strength characteristics of gypsum samples during first days.

Destructive methods, as their name predicates, are possible to be applied on tested specimens only once. Main problem is too many numbers of samples for a description of one problem, e.g. determination of compressive strength on moisture content of samples. A second problem is time, time-consuming preparation before and during own measurements. [7], [8].

II. DESTRUCTIVE METHODS AND PREPARING OF SPECIMENS

The following characteristics were determined using consequently described destructive methods:

- bending strength
- compressive strength
- Young’s Modulus

The compressive strength was tested on six sample halves, obtained after the bending test, which was finished at first. The samples were placed between two steel plates (with dimensions 40×50 mm) in such a way that the lateral edges, which adjoined the longitudinal mould walls during the sample preparation, would be situated on the plate planes. This restricted the effect of the geometry imperfections on the bar surface, which had been cut off. The test itself was made in compliance with the corresponding standard procedures. A typical output of a compressive test is stress-strain diagram.

In second case cylindrical specimens are used for creep tests. Measurement of creep or shrinkage of homogenous material is accompanying by limitations in size of specimens. The length of specimens for testing cement paste creep is depended on the size of gauges. By the optoelectronic probe is possible achieved good results of measuring the creep. Optoelectronic probes are used for measuring of deformation for their high sensitivity on the change of deformation. Their sensitivity is 0.2 μm.

Cylindrical specimens are made into the plastic moulds. Lengths of specimen made in the moulds are between 90 and 100 mm. Specimens with length 70mm are cut from origin length, for tests in creep (Fig. 1). Diameter of all specimens in the moulds is 10 mm. Area for application of load is 78 mm^2.

Strength of hardened gypsum is influenced by type of ground gypsum, fineness of grinding, additives, etc.

The same specimens are used for the creep tests and compression.

Fig. 1: Specimens prepared for creep tests.

All specimens for creep tests were placed before testing in ordinary laboratory environment, where temperature was 20 °C and relative humidity 50 %.

III. INSTRUMENTATION OF THE TESTS

Measuring of the material properties [3] was realized in the MTS Alliance RT 30 tensile testing machine, with scale 30 kN. The three-point bending test was realized with distance of the supports 120 mm (Fig. 2). The signals like a time, load, and deformation of whole specimen were recorded to the PC.

The tensile bending strength was calculated using the standard evaluation procedure as the average of three values. Distance supports, load, dimensions of cross-section are necessary parameters for evaluation procedure.

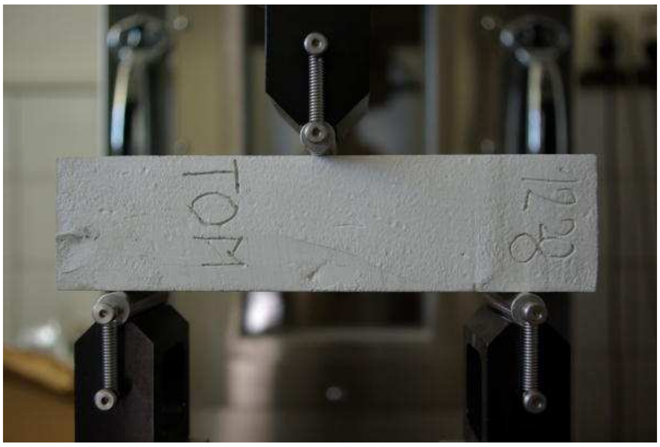


Fig. 2: Specimen in three points bend test.

For evaluation of the compression test was necessary record to the PC signals like a load and strain. Rate of test was defined before its realization. From signals were calculated values of compression strength, which corresponded with time of testing. Goal of measuring was in observe of the compression strength evolution.

Stress, which was calculated from load and loading area and measured strain were signals used for determining stress-strain diagram. From working diagram was calculated Young's Modulus of Elasticity.

Lever mechanism (Fig. 3) [13], equipment for creep measuring and shrinkage of homogenous materials was used for gray gypsum, too. Stationary load is applied to the specimen. A size of the applied load depends on the weight of plumb and location of plumb at the lever. The measuring of deformation is realized by three optoelectronic probes. The length of deformation is whole length of specimen what is placed into the lever mechanism. By the gauges are measured axial deformations. The average deformation is calculated after termination of measuring.

In executed experiments were used specimens with diameter 10 mm. The applied loads were approximately 74N for first sets of specimens. Second one set of specimens was loaded by force 116 N. Force 74N corresponds with weight of equipment above the specimen. The loading at specimens were constant for whole period measuring, which was 37days.

Specimens were firstly placed into the lever mechanism and after their placing were systems loaded by plumbs. Measuring the deformation was start after specimen placing into the lever mechanism. The plumbs were taken off before finishing of measuring, like is possible see in Fig 5 (unloading parts of graphs). After then, all specimens were taken out of lever mechanisms and prepared for compression tests.

In compression tests were checked of the material properties of hardened gypsum.

Fig.3: Lever mechanism – device for testing creep of homogenous materials.

The Modulus of Elasticity and compression strength was computed from data of measuring. Young's Modulus of Elasticity was calculated like a secant, linking the start and value at stress-strain curve which correspond to the 1/3 of the strength.

For measuring of strain was used one extensometer applied on surface of cylindrical specimen, with measurement length 25 mm.

IV. DESTRUCTIVE METHODS - RESULTS

Firstly, specimens were tested from 1st to the 6th month of their hardening by destructive methods. At first, the specimens were tested in three point bend test. In all sets (the moment of testing), three specimens were tested in measuring equipment. Compression tests were realized by using the fragments from bend tests. In Fig. 4 are displayed the results from three-point bend tests.

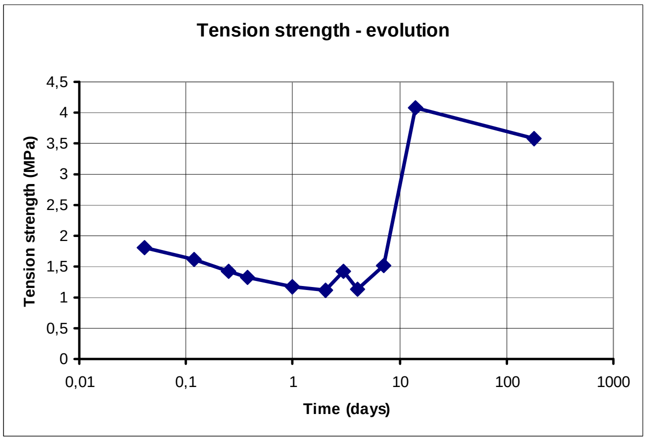


Fig.4: Tension strength and its evolution in time.

In the graph is possible see decreasing trend during the first 3 days. Tension strength is quickly increased between 5th and

14th days. After 14th day are values of tensile strength steady at values from 3.6 MPa to 4.1 MPa. A rapid increasing trend of tensile strength is interesting compare with evolution of weight of prism specimens (Fig. 5).

Fig.5: Evolution of specimen weight in time.

Between 5th and 14th days specimens quickly dry out. Tension and compression strength are increasing (Fig. 6) in the identical time. In the Table 1 are summarized results of compression and tension testing. Strengths are supplemented by standard deviation of sets of data. Measurement in time 1hour and 30minutes after mixing water and gypsum was not realized.

TABLE I
COMPRESSION AND TENSION STRENGTH FROM DESTRUCTIVE TESTING

Time (days)	Compression strength (MPa)	Standard deviation	Tension strength (MPa)	Standard deviation
0.041	3.02	0.526	1.81	0.101
0.0625	3.65	0.088	-	-
0.125	3.01	0.052	1.61	0.122
0.25	2.31	0.348	1.43	0.085
0.375	2.21	0.042	1.32	0.030
1	1.86	0.225	1.17	0.040
2	1.94	0.141	1.12	0.044
3	2.63	0.103	1.42	0.128
4	2.18	0.065	1.13	0.057
7	3.47	0.752	1.51	0.109
14	9.42	0.321	4.07	0.449
180	9.62	0.685	3.58	0.354

Compression strength was steadied between 9.42 MPa and 9.62 MPa in age from 14th to 180th day. From 5th day compression strength steeply increased to 9.42 MPa at 14th day. Compression strength decreased in first days like tension strength (Fig. 5 and Fig. 6).

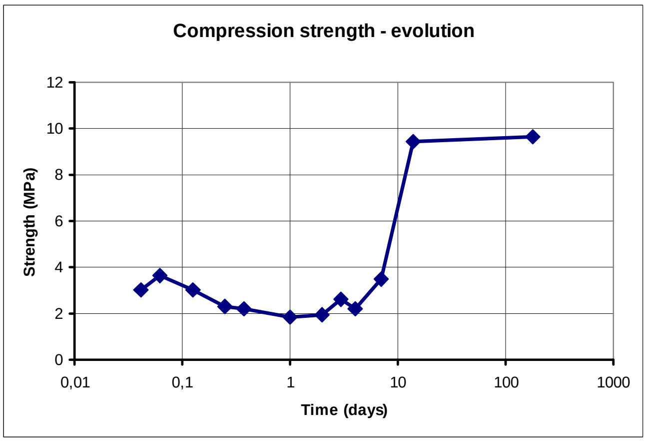


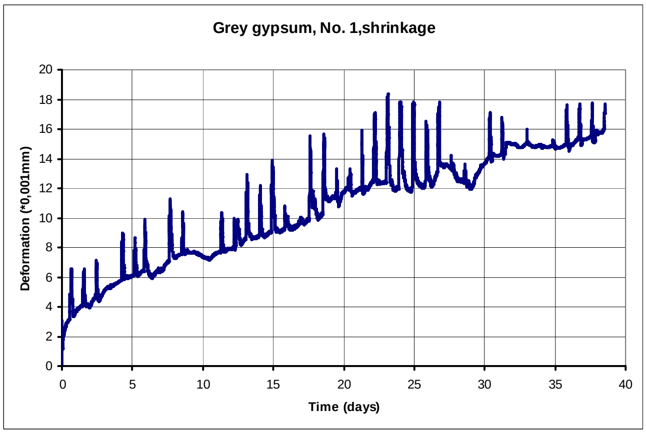
Fig.6: Compression strength and its evolution in time.

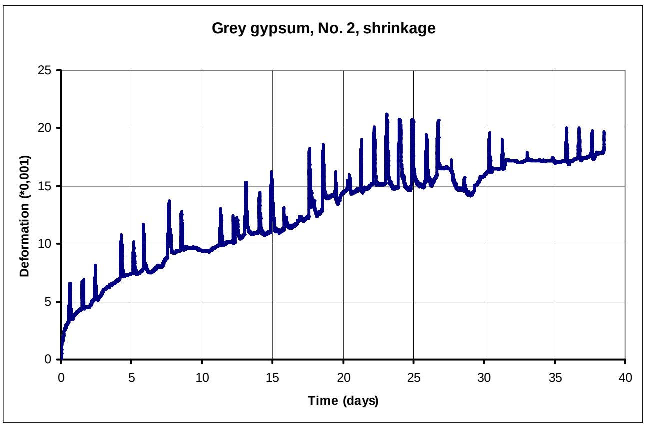
V. CREEP TESTS – RESULTS

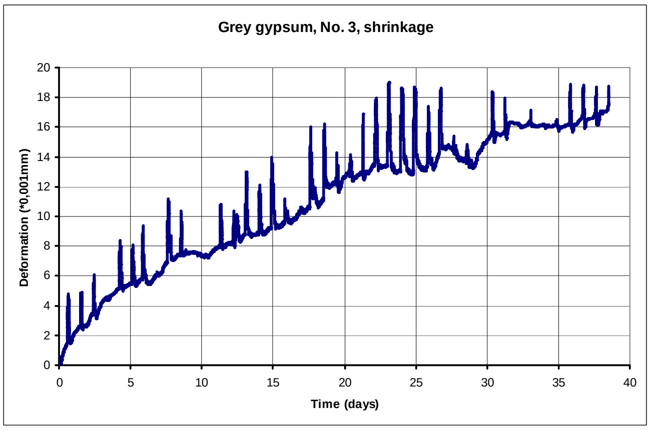
Second set of specimens was tested in lever mechanisms [14]. For testing was used one set, which included 6 specimens. Three specimens were loaded by weight of part of lever mechanism. Next three specimens were loaded by plumbs, placed on the lever. All specimens were 14 days old for time of testing start. Specimens maturing process proceeded in laboratory condition at temperature 20 °C and relatively humidity 50 %. Specimens were covered by plastic wrap before start of creep test.

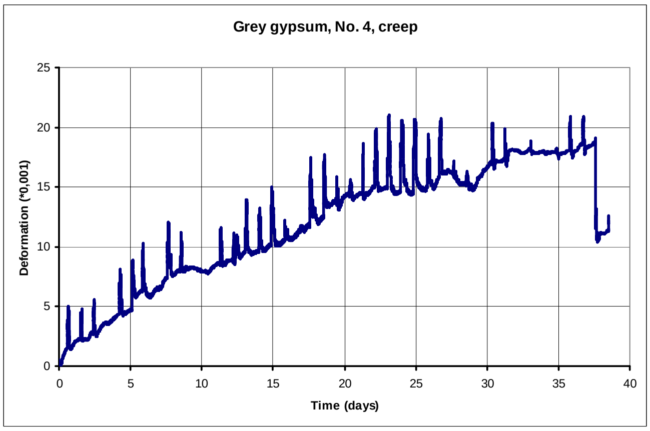
Table II describe deformations from testing of creep and shrinkage. Data are displayed for 37th day after start of tests.

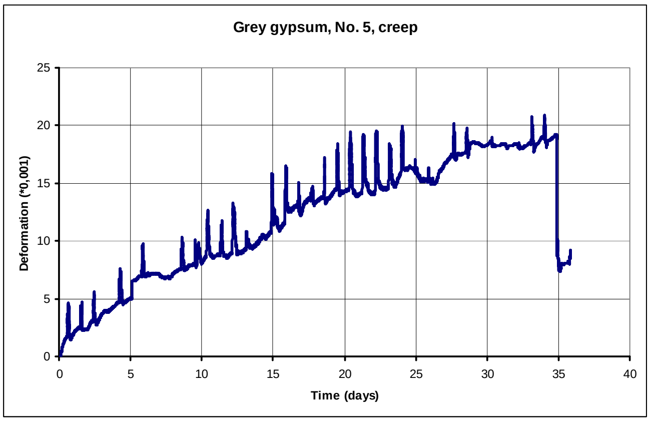
At next graphs displayed the evolutions of specimen's deformation in time (Fig. 7).











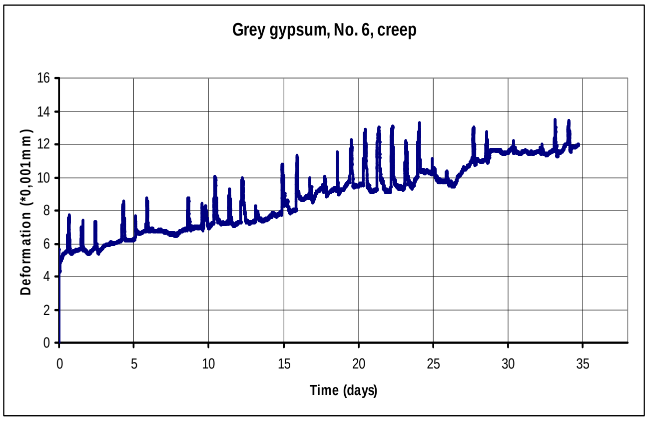


Fig.7: Creep of gray gypsum specimens.

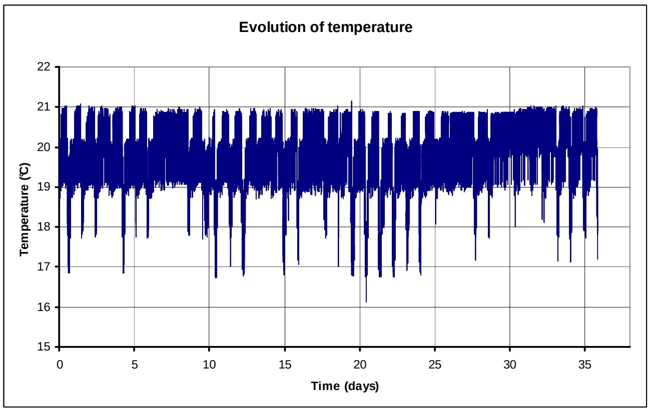


Fig.8: Temperature during the creep test.

In the graphs is viewable trend of increasing the specimen deformation. Rate of increased deformation is steady after 4th day. For higher loaded specimens (No. 4 – 6) was increasing of deformation faster, than for specimens loaded only by weight of lever. In case exclusion of specimen No.6, difference between deformations first and second set is only 3 μm (Table II).

Temperature in laboratory was between 19 and 20 °C. But in Fig. 8 are possible see deviations from steady value of temperature. Temperature deviations were influencing factor for deviations of deformations (Fig. 7).

TABLE II
DEFORMATION OF SPECIMENS AFTER 37 DAYS

Specimen	Deformation (μm)
1	14.7
2	17.0
3	15.9
4	17,5
5	19,2
6	12.4

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VI. CONCLUSIONS

From the results of the static tests, it results that values of mechanical properties of the gypsum started to increase especially after four days of hardening and they stabilized after 14 days. Important influence to the hardening of gypsum is included in saturation of mature gypsum. Paper describes the case when moisture of specimens was non-regulated. Amount of evaporated water was not controlled. Part of water was used to chemical reaction of the gypsum and the second part was evaporated from the material. Process of evaporation of water from hardened gypsum is viewable from Figure 4, 5 and 6. Water leaves the specimen in time and the strength of the material slowly increases to the expected value of the strength of the material.

Compression strength of mature gypsum is approximately 2.5 times higher than strength in bending tension. Trend of relation between strengths is visible during the hardening of gypsum.

Gypsum increases the deformation under constant loading almost linearly. Creep of dry gypsum is measurable and its values are relatively low. Sensitivity of gypsum on changes of temperature is visible from results of creep tests.

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