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ESTIMATION OF MECHANICAL PROPERTIES OF MODIFIED CEMENT STONE BY NANOINDENTING METHOD

The mechanical properties of materials began to appear higher requirements. The development of various technologies has become widespread in the modern world, which has contributed to the creation of new building materials. The nanoindentation method has become an alternative approach to the study of the mechanical properties of materials at the nanoscale. In this case, it is not the dimensions of the sample that are reduced, but the size of the deformed region. During nanoindentation, most solid and superhard non-metallic materials are deformed elastically-plastic, which allows characterizing such mechanical properties as hardness and elastic modulus.

Based on an analysis of literature, it was found that C–S–H exists in at least three different structural forms: low, high, and ultrahigh densities, which have different average values of hardness and hardness and various volume concentrations. The average values of hardness and hardness turned out to be properties inherent in the C–S–H structure, which do not change in various materials based on cement.

In this work, the nanoindentation method was used, preliminary results of the studied cement samples were obtained. The studies were carried out by an automated Hysitron TI 950 TriboIndenter nanoindent. During mechanical measurements, a diamond probe tip was used.

It was found that the elastic modulus M increases in samples that contain a complex additive containing nanosized particles. The effect is also observed when a sol of nanosilica or carbon nanomaterial is introduced into a plasticizing additive. The results are presented by histograms of the distribution of nanoindentation points modulo elasticity E and stiffness H.

Key words: nanoindentation method, cement samples, hardness, elastic modulus, nanoindenter, histogram, phase.

Formulation of the problem. The nanoindentation method was used when a solid needle of a known shape is pressed into the surface of a cement stone sample at a constant speed. When the specified load or indentation depth is reached, the movement stops for a certain time, after which the needle is retracted in the opposite direction. During loading, the values of the load and the corresponding indenter displacement are recorded. The resulting relationship is called the load / unload curve.

Analysis of research and publications, statement of the problem. From this experimental curve, it is possible to determine the hardness and elastic modulus of the material [1-6]. The aim of the study is to study the effect of a complex nanodispersed additive consisting of a superplasticizer and nanoparticles (nanosilica sol and carbon nano-material) on the properties of a cement material by nanoindentation.

Research Methods. Applicable Indenters. When conducting mechanical contact measurements, such as indentation, an important parameter of the testing machine is the hardness of the material from which the probe tip is made. The mechanical strength of the tip should be higher than that of the test material. In this case, the tip retains its shape, and constant test conditions are maintained throughout the experiment. Constant experimental conditions simplify the calculation and description of the experimental results. The most popular material used in such equipment is diamond, as it is the hardest known natural material. But because of the difficulty in processing and shaping, diamond is often replaced with softer materials (for example, aluminum oxide, quartz). Another disadvantage of diamond is its low electrical conductivity, which makes it popular to use solid conductive materials, such as silicon carbide. The high electrical conductivity of such tips allows local measurements of electrical properties.

Nano-mechanical testing device TriboIndenter Brooker TI 950. The Hysitron TI 950 TriboIndenter Nanoindent is an automated high-performance tool to support numerous methods of nanomechanical and nanotribological characteristics. The Hysitron TI 950 nanoindenter system includes the powerful advanced Performech I control module, which greatly improves the accuracy of nano-mechanical testing with feedback, provides dual-head testing for nano / microscale connectivity, and provides unprecedented performance with minimal noise.

Prototypes. The nanoindentation tests were carried out on samples with dimensions of 10 × 10 × 20 mm (3 samples of each composition).

Tests were carried out on 4 formulations:

Sample №. 1 – contains an additive superplasticizer (SP)

Sample № 2 – containing the additive SP + sol nanosilica (NK)

Sample № 3 – containing an additive of superplasticizer SP + carbon nanomaterials (CNM)

Sample №.4 - containing the additive of the superplasticizer SP + sol NK + CNM.

The additive for samples №1-4 was introduced in an amount of 0.8% by weight of cement. The amount of mixing water for all samples was selected in such a way as to obtain a dough of normal density in all cases. Samples were made from cement paste of normal density.

To carry out the experiment, the manufactured samples were ground on a single-disk grinding – polishing machine. Grinding was carried out in order to reduce the surface roughness of the test sample and thus reduce its effect on the final indentation results.

The roughness (rms size of the protrusions and depressions) of the surface of the samples for nanoindentation must be brought to a value of at least 10 nm. This is in order to properly study the heterogeneity in the structure of the C–H–S gel. The characteristic sizes in the structure of the C–H–S gel are 5, 30, and 60 nm. The maximum immersion depth of the nanoindenter is 600-700 nm. The maximum force for the indenter used is 12 mN (milli Newtons).

The following loading procedure was proposed: 1) an increase in force from 0 to 12 mN until maximum immersion is achieved within 10 s; claim 2) for 5 seconds, stop the nanoindenter at maximum depth with a maximum force of 12 mN; p. 3) raising the nanoindenter to the surface, reducing the force to 0 for 10 s.

Improving the nanoindentation technique.

It turned out that point 2 is impossible to fulfill for two reasons: to maintain a constant load of 12 mN for 5 s will not work, because in this case, the depth will probably change due to creep; it is impossible to maintain a constant depth, for example, at 600 nm also because of creep, because the load will decrease.

Immersion depth of the nanoindenter. The size of the inhomogeneities in the structure of the C–H–S gel is 5, 10, and 60 nm. The maximum size of the inhomogeneity is 60 nm. If taken with a 5-fold margin, to distinguish heterogeneity, a maximum depth of immersion of a nanoindenter of 300 nm is required, then for a maximum load it is possible to limit it to 4 mN. For an immersion depth of 200 nm, 1-2 mN is usually sufficient. Considering that the samples showed a compressive strength of 2 times more than usual, a maximum force of 4 mN should be adopted for an immersion depth of 300 nm.

Timing diagram of the load: 1. immersion up to 300 nm and maximum force for 10 s; 2. constant load mode for 5 s; 3. raising the nanoindenter to the surface, relieving the load to 0 within 10 s.

Immersion speed of the nanoindenter: if we take the maximum immersion depth of the nanoindenter to 300 nm, the immersion time is 10 s, then the immersion speed is 300 nm / 10 s = 30 nm / s. Experiment Features:

1. Used type of nanoindenter with Berkovich tip with a cone angle of 143 degrees;
2. In a single test (immersion-exit to the surface) we obtain two standard force-depth curves for loading and unloading;
3. The value of the maximum load and the contact area of the nanoindenter with the sample is calculated hardness H;
4. Using the tangent of the angle of inclination of the tangent to the unloading curve at the upper point, the elastic modulus E at one point is calculated;
5. During the polishing of the samples, a roughness of 12 to 25 nm was achieved.

When the samples reached 28 days of age, it was necessary to determine the test area for each of the 4 samples (selected where there are no pores). These coordinates were entered and stored automatically.

Main part. A complete grid (with 630 points) was drawn for only two samples; for the 3rd, only one line is missing; for the last, 381 instead of 630 points.

After finishing the measurements, the results are presented by histograms of the distribution of nanoindentation points modulo elasticity E and stiffness H and their approximation by 3 Gaussian curves (Figure 1-4).

Phases with their average values of E and H (and moderate statistical scatter within each phase) are highlighted in the histograms. Then, the percentage of each phase was estimated.

Analysis of the results. The procedure for approximation by Gaussian functions: start parameters and the number of phases n are set, and the Matlab program selects the average M and H, the average deviations for each phase, and the volume fractions of the phases from the condition that the sum of the squared deviations between the experimental and theoretical points is minimum.

The preliminary results obtained give a qualitatively correct picture. Very importantly, the distribution modulo elasticity M shifted to the right in samples № 2, 3, 4 in comparison with sample № 1. In terms of stiffness H, there is also a slight shift to the right.

Nanoindentation primarily shows differences in structure, and such significant differences between samples with nanoparticles № 2, 3, 4 and sample № 1 without NPs have already been identified, and these differences should be attributed to the action of nanoparticles [7-10]. This is the only thing that can be reliably established - a shift in the structure between samples № (2, 3, 4) and № 1.

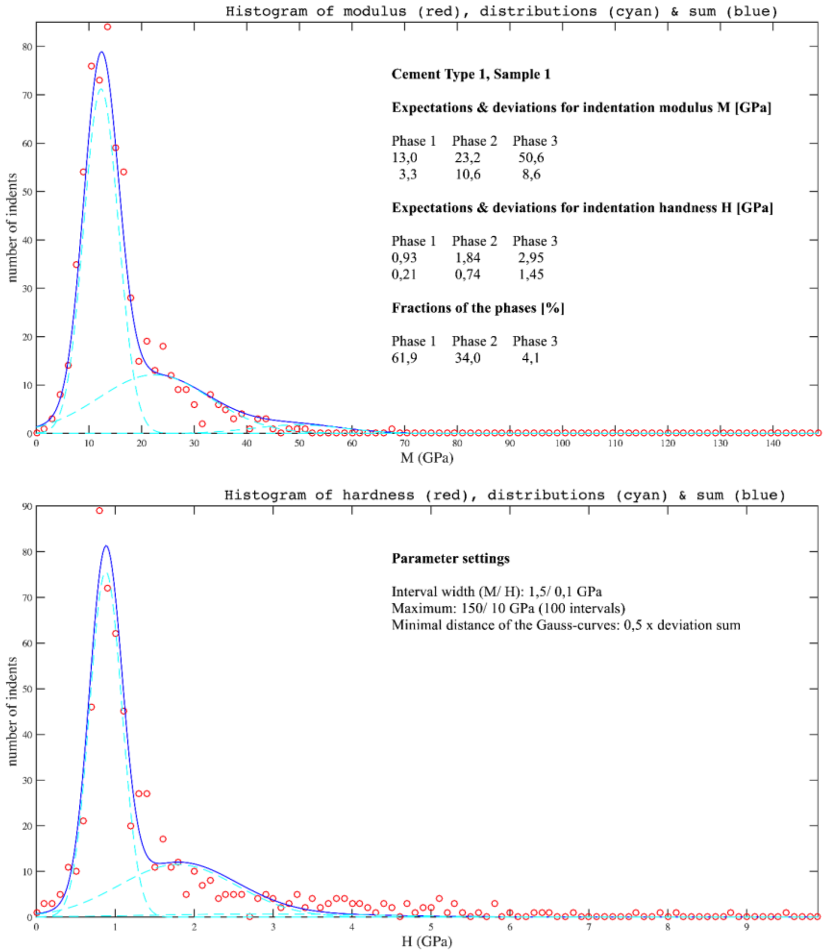

Fig. 1 – Histogram of the distribution of nanoindentation points modulo elasticity E and stiffness H for sample № 1

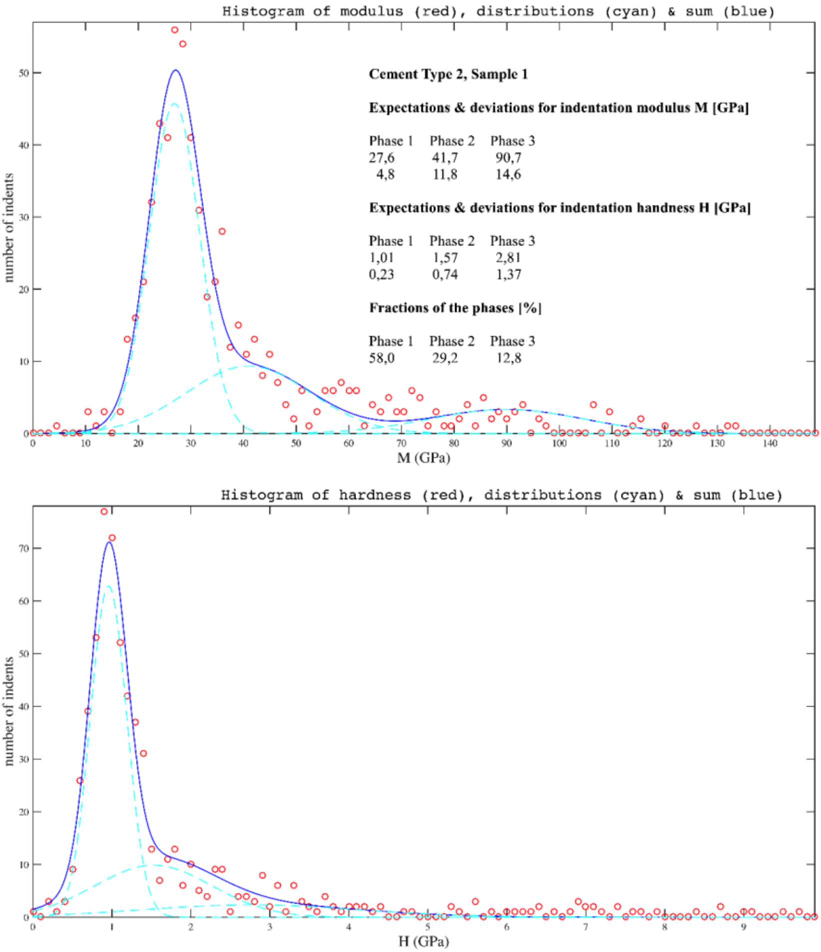

Fig. 2 – Histogram of the distribution of nanoindentation points modulo elasticity E and stiffness H for sample № 2

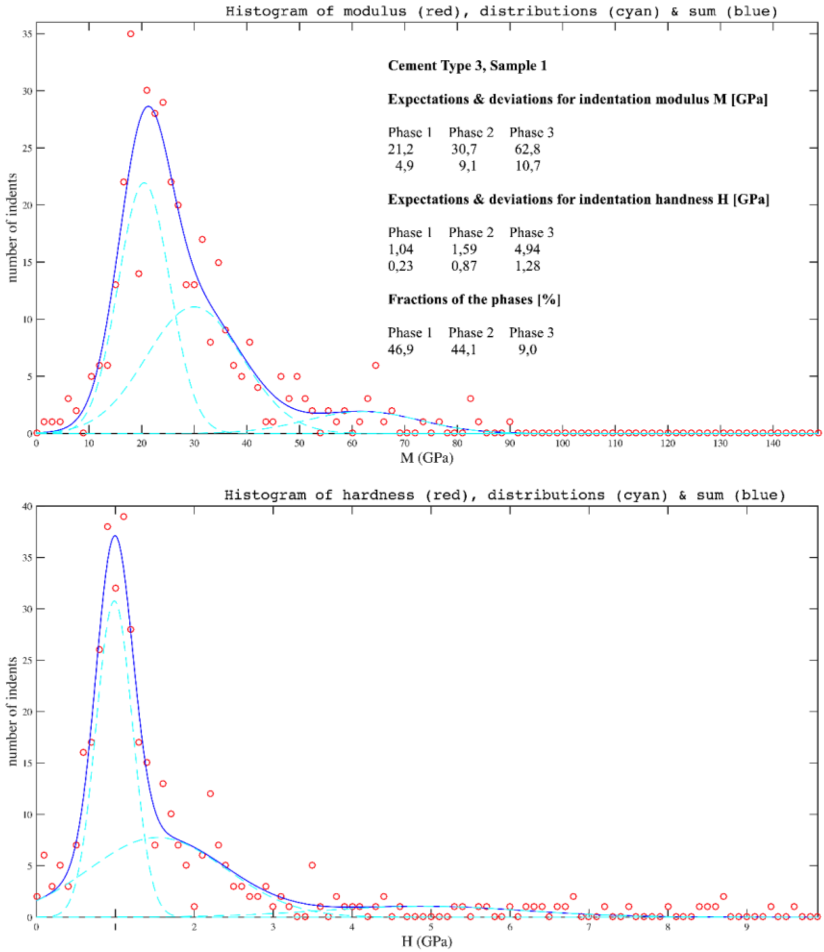

Fig. 3 – Histogram of the distribution of nanoindentation points modulo elasticity E and stiffness H for sample № 3

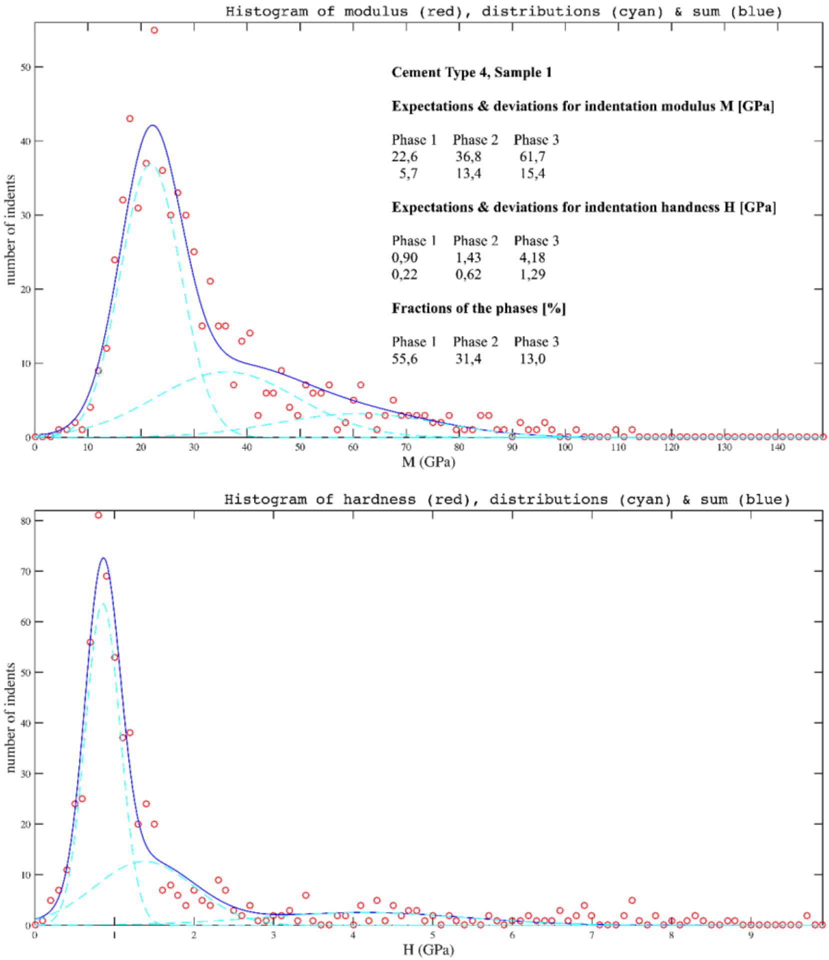

Fig. 4 – Histogram of the distribution of nanoindentation points modulo elasticity E and stiffness H for sample № 4

Conclusions:

For all samples, we have a large test series of 600 indents, followed by a smaller series of 135 indents each. Reproducibility is satisfactory, and there is a tendency to decrease the average values of M and H from sample № 4 to 1. Deconvolution will be a difficult task, but it can be solved.

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Е.Н. Полонина, С.Н. Леонович

Оценка механических свойств модифицированного цементного камня методом наноиндентирования

К механическим свойствам материалов стали появляться более высокие требования. Развитие различных технологий получили широкое распространение в современном мире, что способствовало созданию новых строительных материалов. Метод наноиндентирования стал альтернативным подходом к исследованию механических свойств материалов на наноуровне. В этом случае уменьшаются не размеры образца, а размер деформированной области. При наноиндентирования большинство твердых и сверхтвердых неметаллических материалов деформируется упруго-пластически, что позволяет характеризовать такие механические свойства, как твердость и модуль упругости.

На основе анализа литературных источников выявлено, что C–H–S существует, по крайней мере, в трех различных по структуре формах: низкой, высокой и сверхвысокой плотностях, которые имеют различные средние величины жесткости и твердости и различные объемные концентрации. Средние величины жесткости и твердости оказались свойствами, присущими структуре C–H–S, которые не меняются в различных материалах, основанных на цементе.

В данной работе использован метод наноиндентирования, получены предварительные результаты исследуемых цементных образцов. Исследования осуществлялись автоматизированным наноиндентом Hysitron TI 950 TriboIndenter. При проведении механических измерений использовался алмазный наконечник зонда.

Установлено, что модуль упругости М увеличивается у образцов, которые содержат комплексную добавку, содержащую наноразмерные частицы. Эффект также наблюдается и при введении в пластифицирующую добавку, как золь нанокремнезема, так и углеродный наноматериал. Результаты представлены гистограммами распределения точек наноиндентирования по модулю упругости Е и жесткости Н.

Ключевые слова: метод наноиндентирования, цементные образцы, твердость, модуль упругости, наноиндентор, гистограмма, фаза.

О.М. Полонина, С.М. Леонович

Оцінка механічних властивостей модифікованого цементного каменю методом наноіндентування

До механічних властивостей матеріалів стали з'являтися більш високі вимоги. Розвиток різних технологій набули широкого поширення в сучасному світі, що сприяло створенню нових будівельних матеріалів. Метод наноіндентування став альтернативним підходом до дослідження механічних властивостей матеріалів на нанорівні. В цьому випадку зменшуються не розміри зразка, а розмір деформованої області. При наноіндентування більшість твердих і надтвердих неметалічних матеріалів деформується пружно-пластично, що дозволяє характеризувати такі механічні властивості, як твердість і модуль пружності.

На основі аналізу літературних джерел виявлено, що C–H–S існує, принаймні, в трьох різних за структурою формах: низькою, високою і надвисокою щільності, які мають різні середні величини жорсткості та твердості й різні об'ємні концентрації. Середні величини жорсткості та твердості виявилися властивостями, притаманними структурі C–H–S, які не змінюються в різних матеріалах, заснованих на цементі.

У даній роботі використаний метод наноіндентування, отримані попередні результати досліджуваних цементних зразків. Дослідження здійснювалися автоматизованим наноіндентом Hysitron TI 950 TriboIndenter. Під час проведення механічних вимірювань використовувався алмазний наконечник зонда.

Встановлено, що модуль пружності М збільшується у зразків, які містять комплексну добавку, що містить нанорозмірні частинки. Ефект також спостерігається і при введенні в пластифікуючі добавку, як золь нанокремнезема, так і вуглецевий наноматериал. Результати представлені гістограмами розподілу точок наноіндентування по модулю пружності Е і жорсткості Н.

Ключові слова: метод наноіндентування, цементні зразки, твердість, модуль пружності, наноіндентор, гістограма, фаза.

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