

Study of the carbon nanomaterials synthesis on the cement binder

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Abstract: The authors have developed a technology for creating a nanomodified binder for construction purposes, in which a uniform distribution of carbon nanomaterials is achieved by controlled synthesis of nanomaterials in the structure of the binder. The synthesis of carbon nanomaterials (CNM) was carried out by chemical vapour deposition of hydrocarbons on metal oxide catalysts. Cement CEM I 42.5H (M500 D0) and metal-oxide catalyst Ni–MgO (92.5 %–7.5 %, obtained by thermal method) were used as a raw material. The optimum catalyst/cement ratio of 0.2 was found to yield the specified CNM quality. If the catalyst/binder ratio is increased (without changing the other parameters), the specific yield value of the nanomodifier decreases, which can be explained by “poisoning” of the catalyst and, consequently, the formation of unstructured carbon. The nanomodifier was investigated by scanning electron microscopy, Raman spectroscopy and thermal gravimetry. Using Raman spectroscopy, the shape and position of the characteristic G and D bands showed that a material containing multi-walled carbon nanotubes (MWCNTs) was synthesized on the cement binder matrix. According to thermal gravimetry data, the obtained nanomodifier is resistant to thermal decomposition up to 500 °C. Experimental studies of the influence of the obtained additive on the characteristics of the construction composite were carried out on samples of fine-grained concrete. It is established that compressive strength for nanomodified samples increases by 18–20 %.

Keywords: carbon nanotubes; nanomodifier; cement binder; chemical vapour deposition method; compressive strength.

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Исследование процесса синтеза углеродных наноматериалов на цементном вяжущем

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Аннотация: Разработана технология создания наномодифицированного вяжущего строительного назначения, в котором равномерное распределение углеродных наноматериалов достигнуто за счет контролируемого синтеза наноматериалов в структуре вяжущего материала. Синтез углеродных наноматериалов (УНМ) осуществляли способом газофазного химического осаждения углеводородов на металлоксидных катализаторах. В качестве исходного сырья использован цемент ЦЕМ I 42,5Н (М500 Д0) и металлоксидный катализатор Ni–MgO (92,5 %–7,5 %, полученный термическим методом). Для получения заданного качества УНМ найдено оптимальное соотношение катализатора к цементу – 0,2. При увеличении соотношения катализатор / вяжущее (без изменения остальных параметров) значение удельного выхода наномодификатора снижается, что можно объяснить «отравлением» катализатора и, как следствие, образованием неструктурированного углерода. Наномодификатор исследован с помощью методов сканирующей электронной микроскопии, рамановской спектроскопии и термогравиметрии. С помощью рамановской спектроскопии по форме и положению характеристических полос G и D установлено, что на матрице цементного вяжущего синтезирован материал, содержащий в своем составе

многослойные углеродные нанотрубки (МУНТ). По данным термогравиметрии полученный наномодификатор устойчив к термическому разложению до 500 °С. Экспериментальные исследования влияния полученной добавки на характеристики строительного композита проводились на образцах мелкозернистого бетона. Установлено, что прочность на сжатие для наномодифицированных образцов увеличивается на 18–20 %.

Ключевые слова: углеродные нанотрубки; наномодификатор; цементное вяжущее; метод газофазного химического осаждения; прочность на сжатие.

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1. Introduction

Presently, big research interest in the development and practical application of carbon nanostructures is driven by their high performance properties, such as exceptional tensile strength, high elastic modulus and a number of other important indicators, which are key aspects in the formation of composite materials for special purposes. The most difficult stage in the application of nanomodifying additives is their uniform distribution in the composite matrix [1]. Due to their high agglomeration capacity, they cannot be evenly dispersed, and multi-stage and labour-intensive processes are required to overcome these factors. The solution of such problems will open up additional opportunities to create specialised composites with improved qualitative and functional characteristics through the use of nanostructuring systems in the material matrix. Such an approach, in turn, will be a constructive help for industries which manufacture products under special conditions of high intensity or where the end product itself is to be exploited under unfavourable climatic conditions and other limiting factors [1].

Studies [1–12] discuss the issues of synthesis of nanomodifiers on matrix components of composites (cement, sand, gypsum, zeolite, etc.) and consider the characteristics and growth stages of particles and new phases of the nanoscale range.

A method of organizing uniform distribution of carbon nanotubes and nanofibers by their growth on the surface of the matrix particles was proposed in [1]. Silica and cement particles were used as a matrix. The growth on the initial composite components provides an important advantage of such dispersion by forming a stable and sustainable bond between the nanomaterials and the matrix particles. The actual synthesis process was carried out by chemical vapour deposition (temperature from 550 to 750 °C) at atmospheric pressure, using a gas mixture based on acetylene and methane. Cement components were used without prior treatment because the material contains iron oxide, which can act as a catalyst. For the silica the source of the catalyst was iron

nitrate followed by impregnation. In all the samples synthesised, the presence of carbon nanomaterials of 30 to 50 nm and structures of 3 to 15 µm in length was confirmed [1].

In [2], features of nanocarbon synthesis from naturally occurring materials (peat of Sogra deposit) were analysed. The reaction was carried out by thermal treatment at temperature 505–520 °C (from 40 min to 2 h) with limited oxygen access. Electron microscopic studies confirmed the presence of nanocarbon form in the synthesized samples. A strength check of cement stone modified with nanomodificator on a matrix of natural peat showed an increase from 13.5 to 40 % with an additive concentration of 0.5 % of cement mass.

The effect of synthesized fillers based on aluminosilicates was considered in [3] and the properties were evaluated on cement systems. Synthesis of aluminosilicates consisted of several stages: precipitation from aluminum sulfate $\text{Al}_2(\text{SO}_4)_3$ solution; addition of sodium silicate and washing of precipitate with water. X-ray phase analysis revealed a predominance (78 %) of nanosized crystallite aggregates in the synthesized sample. The setting time of the cement paste containing the synthesized aluminosilicates accelerated by a factor of 3 to 4.

The paper [4] proposes a method for the synthesis of barium hydrosilicates nanobjects, the application of which is aimed at improving the quality of hydration-curing composites. Solutions of barium hydroxide and silicic acid obtained by the interaction of sodium hydrosilicates with iron hydroxide sol were used for the synthesis of barium hydrosilicates. Solutions were found to be stable during storage; no signs of coagulation or gelation were recorded. Researchers suggest the use of nanomodifier as a mechanism to influence or regulate composite structure formation processes.

The processes of mechanochemical activation in the presence of the modifier on Portland cement were studied in [5]. It was established that as a result of combined stepwise milling of the mentioned

components, a structured modifier with nanosized shell was formed, and this material was named as nanocement. The distinguishing structure of such material is the presence of a continuous shell on the cement grains, the thickness of the coating reaching several tens of nanometres (30–60 nm). The functional groups of the applied modifier interact with the calcium and oxygen active centres on the surface of the clinker particles, which contributes to the formation of a continuous nano-shell and generates a composite with high building and technical properties.

In paper [6] options of silica (SiO_2) synthesis by using sol-gel technology process were presented. The reaction of obtaining nanomaterials was based on the principle of polymerization through the growth of metal-oxopolymers in solutions. The choice of silica matrix as an object of study was based on the fact that the substance in question is a related structure to building composites and is actually present in cement binder clinker minerals ($3\text{CaO}\cdot\text{SiO}_2$, $2\text{CaO}\cdot\text{SiO}_2$); as a result of hydrolysis and hydration reactions the synthesized nanoparticles are incorporated into the material structure without restrictions. The synthesized silica nanoparticles present a stable spherical form with a dispersity of 5–10 nm and material stability for 7 days, while the overall system of $\text{SiO}_2\text{--H}_2\text{O}$ modifier contributes to rational introduction and uniform distribution of the composite in the cement matrix.

Nanomodifying agents considered in the literature differ primarily in the principles and mechanisms of synthesis, which directly affect the formation of additive characteristics: size, morphology, specific surface area, particles' interaction energy, as well as the effective dosage degree [7–12]. In terms of efficiency of nanomodifiers [7], varieties of synthesized materials with predominantly spherical, tubular and cylindrical structure prevail. For a composite, this structure of nanoparticles in the form of active additives will fill the micropores (or shrink them) but also reduce the amount of capillary and free mixing water (which has not entered into the interaction reaction) due to sorption processes, thereby obtaining a compacted layout system. It should be noted that in almost all of the reviewed works preference is given to materials that are similar or related in structure to the mineral constituents of cement binder on the principle of physical and chemical structure. This is due to the ability to participate in reactions through topochemical mechanism, building a system of

crystal structure of nanoscale size. The need to find new types of additives, including nano-additives, remains very urgent.

The use of nanomodifiers with a similar structure, such as silica particles [8, 9], is complicated by the short period of effect, in particular the role of the crystallisation centre, which is most often observed in the early periods of hardening (1 day) and is not fixed in later periods. On the other hand, when modifiers based on nanocarbon materials of different structures are applied, their presence is observed throughout the interval of composite formation from the beginning of setting to later terms of the material life cycle.

In this regard, we can conclude that obtaining a nanomodified binder based on a cement matrix with carbon nanofibers embedded in its structure is practically important, as the resulting additive will have the characteristics of the initial system and stable nanostructures.

In this paper a method of obtaining carbon nanostructures in the binder matrix (cement) by controlled synthesis of nanoparticles by chemical vapour deposition is considered. This approach will ensure reliable bonding and strong connection between carbon nanostructures and matrix elements, which is the key criterion for obtaining a homogeneous nanomodified composite.

2. Materials and Methods

2.1. Materials

Cement CEM I 42.5H (M500 D0) (Eurocement, Belgorod, Russia) was used as feedstock; quartz sand from Tambov deposit (JSC Tambov Karieroupravlenie) (Russian Standard 8736–93, average particle size modulus $M_{cr} = 1.34$, true density – $2.63 \text{ kg}\cdot\text{L}^{-1}$, humidity – 1.73 %, group – fine first class); powdered metal oxide catalyst Ni–MgO (92.5 %–7.5 %, production LLC “NanoTechCenter”, Moscow, Russia; propane-butane mix 204. Tambov, Russia); propane-butane mixture Russian Standard 20448–90; noble gas – argon Russian Standard 10157–79.

2.2. Methods of synthesis

The process of nanomodifier synthesis on a binder matrix was carried out at the production site of NanoTechCenter LLC using an industrial line for production of carbon nanomaterials by chemical vapor deposition (Fig. 1). Pyrolysis temperature was $(650 \pm 20)^\circ\text{C}$, synthesis time – 90 min.

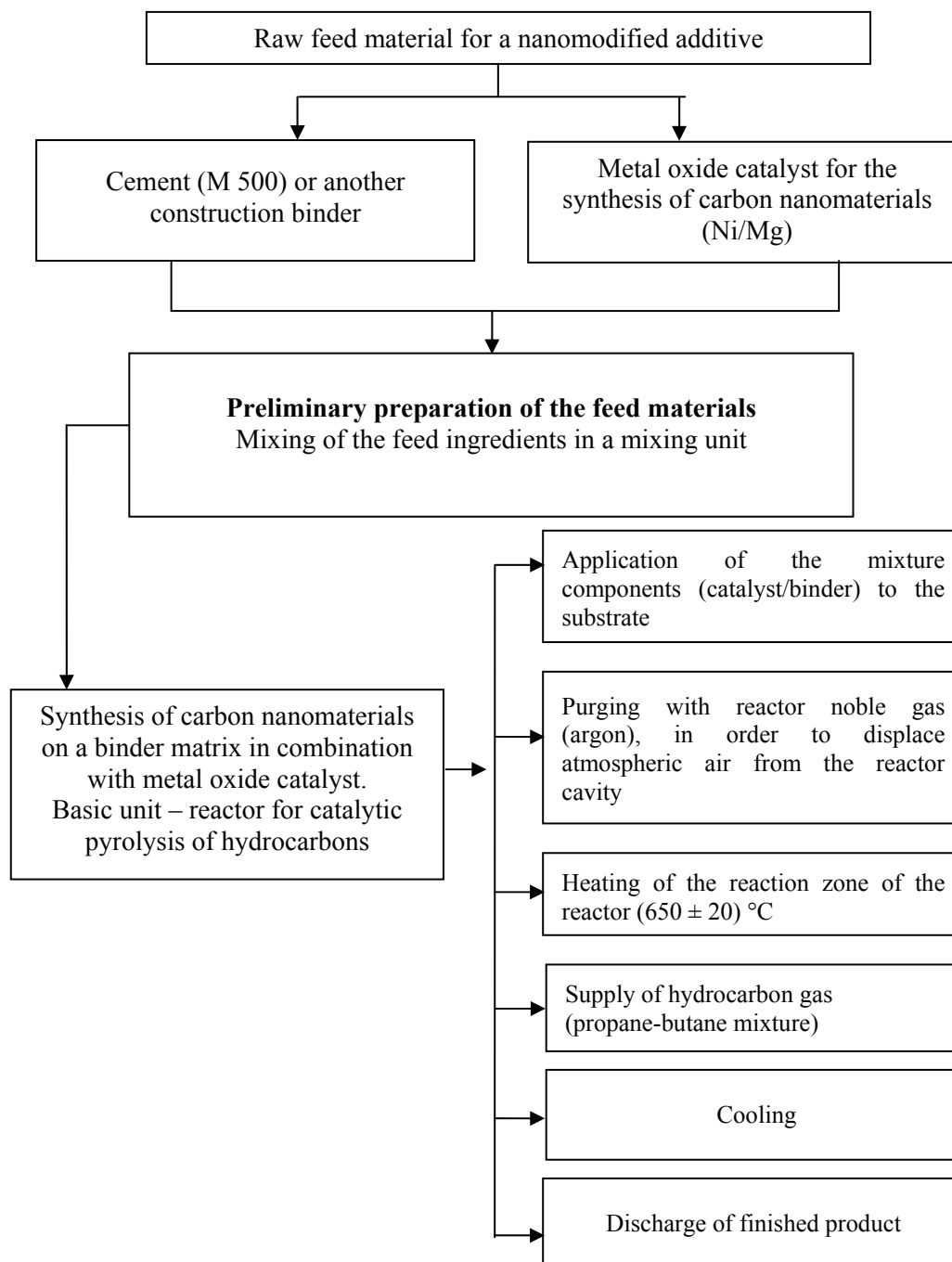


Fig. 1. Schematic diagram of the method of nanomodifier production by synthesis of carbon nanomaterials on the binder matrix

2.3. Characterization methods

The surface morphology of the nanomodifier was studied by scanning electron microscopy (SEM) on a Merlin unit (Carl Zeiss, Jena, Germany). The thermal stability of the sample was determined by thermal gravimetric analysis (TGA) on a STA 449 F3 Jupiter (NETZSCH-Gerätebau, GmbH, Germany). DXR Raman Microscope (Thermo Fisher Scientific, USA) was used to determine the degree of ordering of the crystal structure.

2.4. Structural tests

Experimental studies aimed at investigating the effect of nanomodified additive on the characteristics of the construction composite were carried out on samples of fine-grained concrete. To obtain a construction composition, 704 g of cement and 1,056 g of sand were taken and tap water was added (water/cement ratio of 0.53). The content of nanomodifier varied – 1, 1/2, 1/4, 1/8, 1/16 of the total mass of cement. Mixture was placed in prismatic

mold (40×40×160 mm). All samples of the same series were tested at an estimated age of 28 days. For mechanical tests a testing machine IP-500M-auto (ZIPO, Armavir) was used.

3. Results and Discussion

The synthesis of a nanomodifier based on the principle of catalytic pyrolysis implies that a number of regime parameters affecting the morphology and structure of the synthesized material are dependent. One of the main criteria is the choice of the required amount of the catalyst in the ratio with the cement component in order to maximise the reaction rate and determine the limiting specific yield of the carbon nanomaterial. An optimum catalyst/binder ratio will improve the quality performance of the resulting nanomodifiers by eliminating variants containing unstructured nanocarbon particles.

Experimental studies (Fig. 2) to determine the catalyst/cement ratio ranged from 0.1 to 1 with a discreteness of 0.1. It was found that by increasing the catalyst/binder ratio (without changing other parameters), the specific yield value of the nanomodifier decreases. This can be explained by "poisoning" of the catalyst, an overabundance and complete deactivation of the catalyzing phase, which leads to the formation of unstructured carbon or pyrocarbon in high concentration without maintaining the homogeneity of the resulting product. Thus, it was found that the highest specific yield of nanoparticles is observed at a ratio of 0.2.

According to the SEM results, the synthesized carbon nanomaterials are nanofibers (Fig. 3c, f), presumably multi-walled carbon nanotubes (MWCNTs). During catalytic pyrolysis, the formation of MWCNTs is observed not only in the

samples with the catalyst in the binder, but also on the grains of the original cement, which was treated in the reaction zone of the chamber (Fig. 3b, e). The nanostructures in the cement samples (without catalytic mass) are attributed to the presence of iron oxide in the binder, which acts as a catalyst and substrate for the synthesis of MWCNTs. The particle size of the catalyst (NiO/MgO) and the average cement grain size lie in a close range of values. However, looking at the micrographs (Fig. 3b, e), it can be concluded that the amount of initial iron oxide for the synthesis of nanostructures was insufficient. At the same time, the complex application (NiO/MgO + Fe₂O₃) forms a single chain of nanostructures due to the parallel synthesis on different carriers.

According to Fig. 3f, catalyst particles are located at the ends of the MWCNTs, which are characterized by a predominantly apical-type growth pattern. Small amounts of amorphous nanocarbon (including those on the surface of the nanomaterial) in the form of various sized carbon clusters of a non-tubular structure can also be observed.

The quality assessment and identification of the regularities in the structural parameters of the nanomodified binder was carried out by Raman spectroscopy (Fig. 4). In the composition of the nanomodified cement, clear peaks are present indicating the nanotube content in the samples – G-peak at $\approx 1594\text{ cm}^{-1}$ and D-peak at $\approx 1339\text{ cm}^{-1}$ [13]. The G-peak has a sharp angular structure without significant visible peak bifurcation, in contrast to semiconductor-type nanotubes which exhibit splitting of the upper part of the spectrum. The multilayer structure of MWCNTs is confirmed by the absence of the RBM in the low frequency range ($\approx 180\text{--}250\text{ cm}^{-1}$). Similar regularities are not observed in the spectra of the initial binder.

The appearance of strong peaks in the range $850\text{--}700\text{ cm}^{-1}$ (Fig. 4) in the materials of the initial composition and the nanomodifier refers to full-symmetric valence oscillations of predominantly polyhedral structure. This mode is very sensitive to structural distortions and to the presence of local disordering, both in the cationic and oxygen sublattice.

The absence of the considered peak in the sample of nanomodifier may be attributed to rearrangement and growth of newly formed symmetrical structures due to the synthesis process and increase in chemical bond length. Also, disappearance of the $850\text{--}700\text{ cm}^{-1}$ peak in the nanostructured binder may be a result of the influence of oxygen and cation vacancy filling processes in the

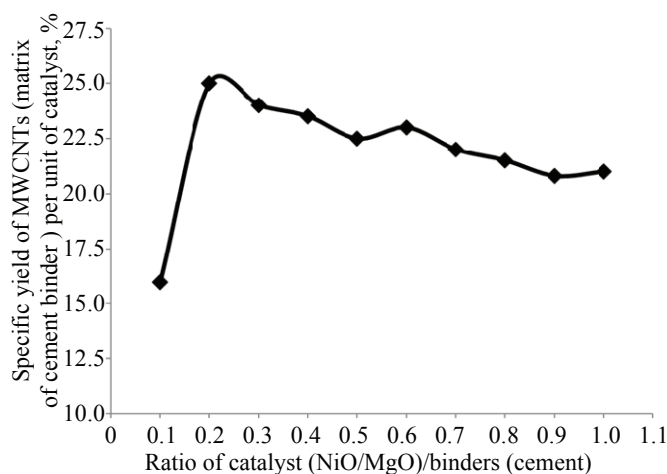


Fig. 2. Determination of the catalyst (NiO/MgO)/binder (cement) ratio

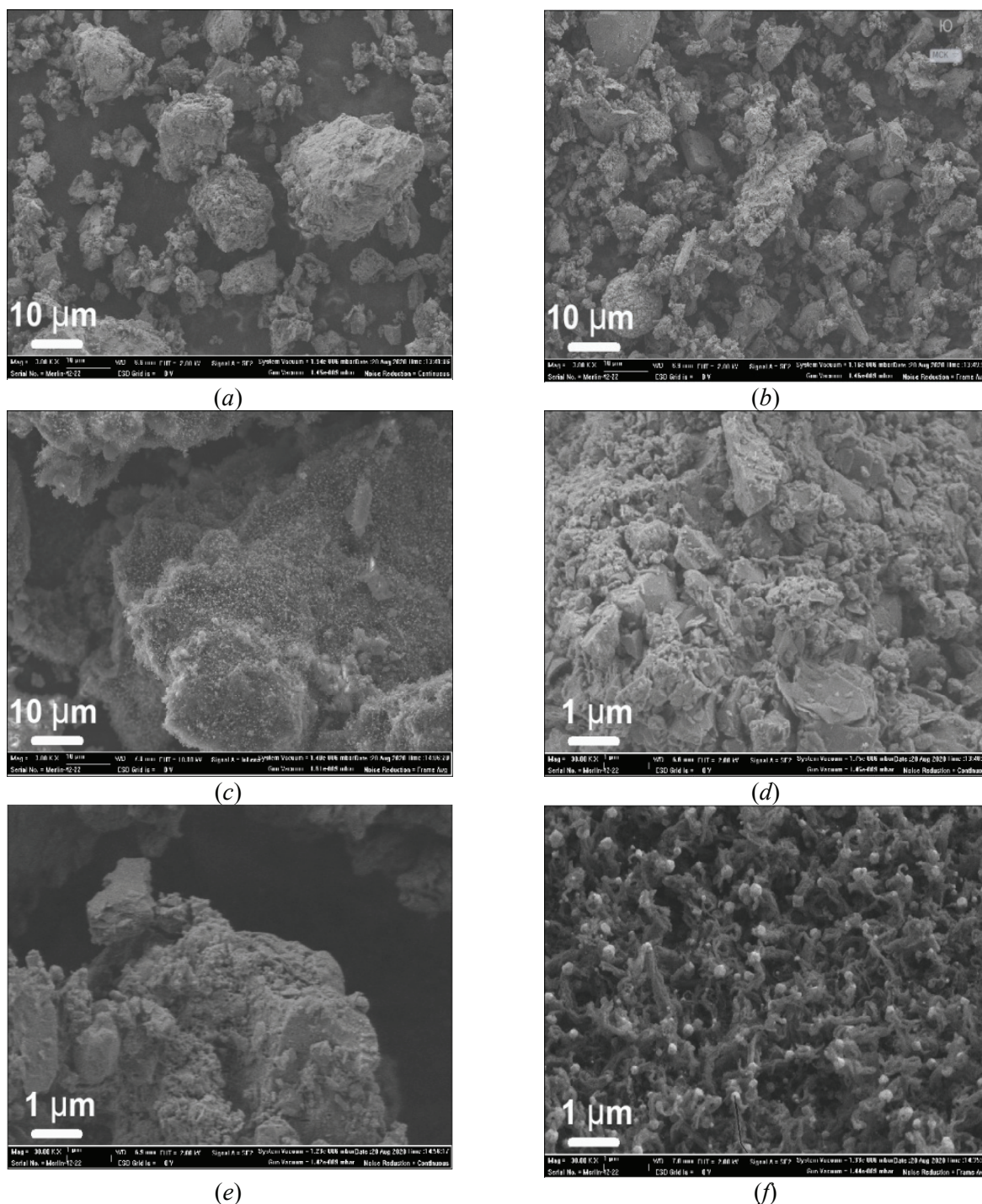


Fig. 3. SEM images of the synthesized materials' structure:

a, d – initial composition of the cement binder; *b, e* – cement binder after processing in the MWCNT synthesis chamber; *c, f* – nanomodifier on the cement binder

atom sublattice. Accordingly, in the spectra of modified samples the intensity of $850\text{--}700\text{ cm}^{-1}$ peak either decreases – in case of partial filling of oxygen vacancies, or this peak practically disappears – in case of full filling of oxygen vacancies available in the structure [10, 13].

The thermal stability of carbon nanomaterials depends directly on the structural features and the

presence of defects (e.g., purity and homogeneity). A material is considered thermally stable if its chemical structure or physical properties remain stable at elevated exposure temperatures [14, 15]. Thermal gravimetric analysis makes it possible to determine residual products of chemical reactions and to evaluate the degree of stability of substances during heat treatment. Carbon (in any form) reacts

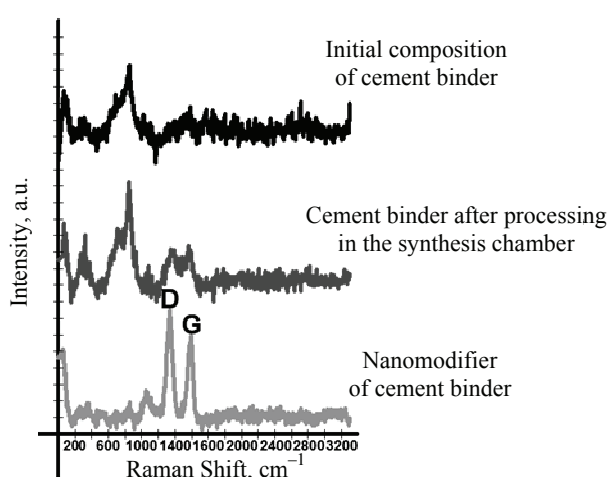


Fig. 4. Raman-spectrum of the initial composition and the synthesized samples

with oxygen to form carbon dioxide (CO_2). The main factors affecting the oxidation reaction are the specific surface area and the defect level of the material. An increase in the specific surface area or defectiveness leads to an intensification of the oxidation reaction and therefore a reduction in the oxidation limit temperature [14].

According to the thermal gravimetric data, the initial nanomodifier (Fig. 5c) is resistant to thermal degradation up to temperatures of 440–460 °C. The mass loss of the sample starts at 500 °C and continues up to 540–640 °C. As can be seen from the thermal gravimetric analysis curves (Fig. 5), the mass loss for the original cement was about 0.5 %, for the CNT synthesis chamber-treated cement less than 6 %, and for the nanomodified cement almost 26 %. The change in mass values is related to the process of thermo-oxidative degradation of the material, which leads to the sublimation of metal catalyst particles, including encapsulated ones [16]. The presence of amorphous forms in the nanomodified sample has not been established because in this state carbon is the least stable to oxidation and begins to oxidize above the temperature of 300 °C. MWCNTs exhibit oxidation reactions like other materials of carbon origin. Depending on the specific surface area and the forming graphite layers, they react in an oxidizing environment at 400–600 °C [17]. The gradual decrease in mass at 300 °C in a sample of cement treated in the reactor chamber indicates the presence of some amorphous carbon phase.

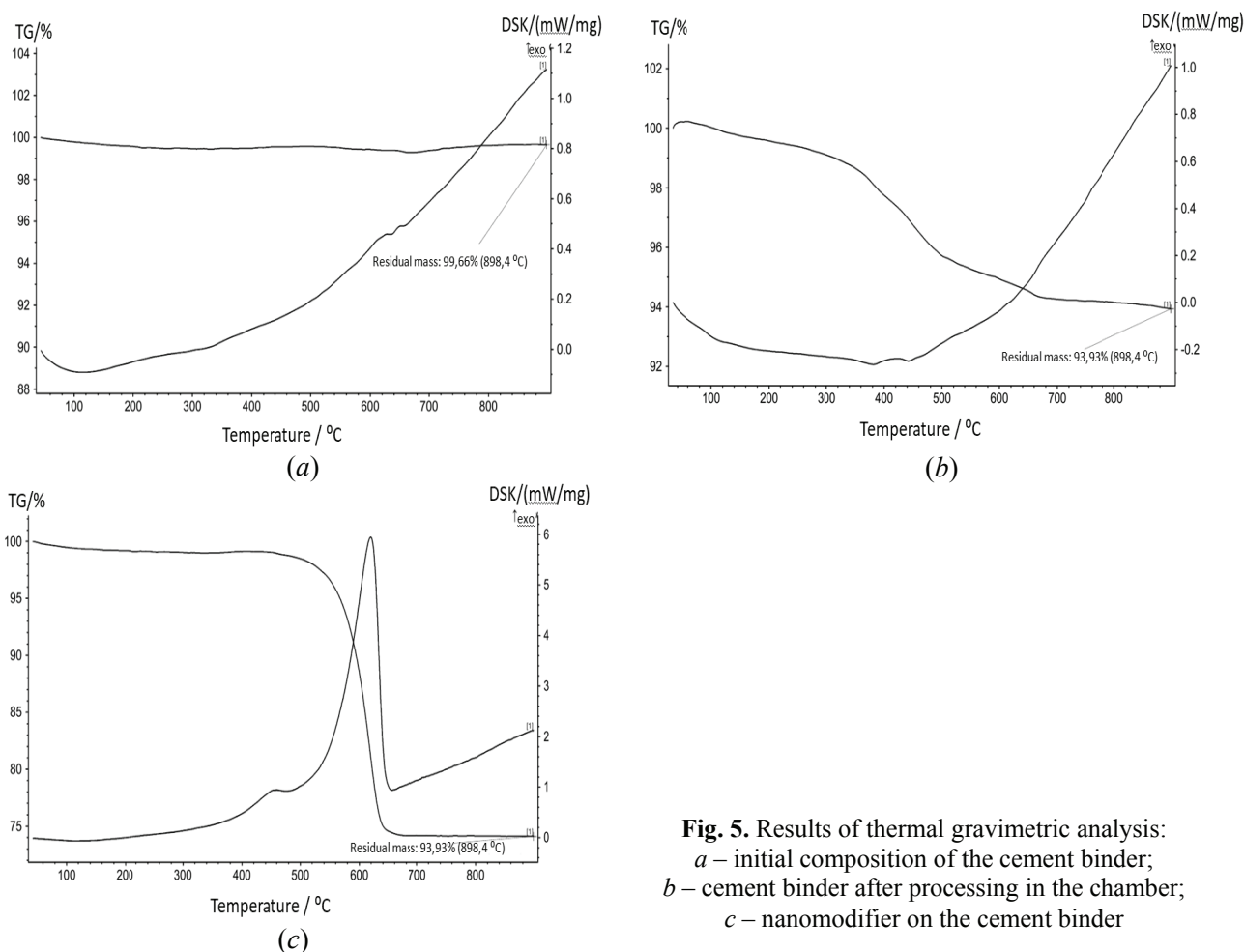


Fig. 5. Results of thermal gravimetric analysis:
 a – initial composition of the cement binder;
 b – cement binder after processing in the chamber;
 c – nanomodifier on the cement binder

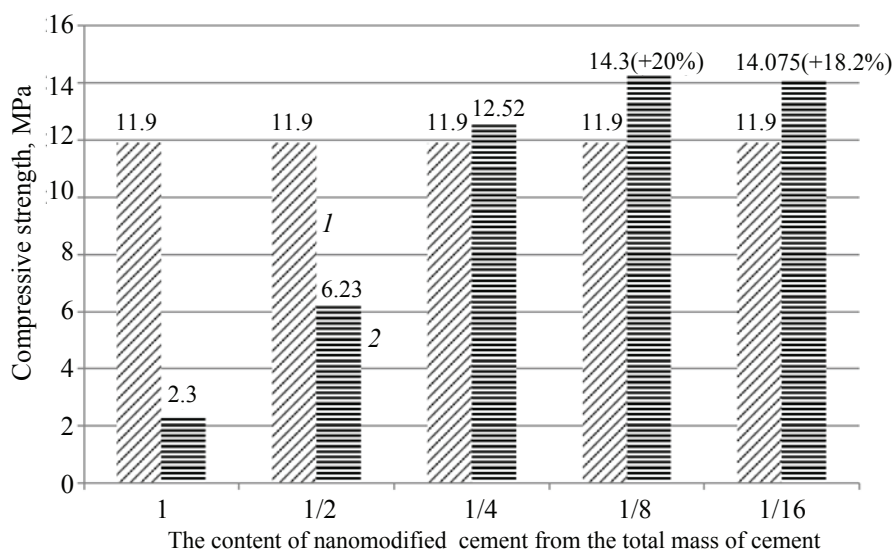


Fig. 6. The effect of nanomodified cement on the compressive strength of fine grain concrete:
1 – non-modified; 2 – nanomodified

The production of the nanomodifier can be regarded as an intermediate preparatory step in the formation of a construction composition consisting of clusters of new phases and crystals. Hydration of cement predominantly develops as a heterogeneous physical and chemical process, proceeding with the participation and great influence of cement grain surface factor – surface area and its properties [10, 11, 18, 19]. Therefore, nanomodifying the binder will lead to chemical activation of the surface of cement particles and will contribute to physical and chemical change of surface properties due to the development of a new phase and newly formed nanostructural level.

The proposed technology targets the production of construction materials and modification of various kinds of binders. The resulting effect of using a modifying additive is an increase in the physical and mechanical characteristics of the construction composite. The results of strength tests showed a steady increase in the physical and mechanical properties of the material.

It has been established, that compressive strength for samples with nanomodifying additive in quantity 1/8–1/16 is increased on 18–20 % in comparison with control samples of a construction composite.

4. Conclusion

The paper proposes a technology for obtaining a nanomodifier on a binder matrix by chemical vapour deposition. The process of nanostructuring of composite materials using such technology can be divided into two interrelated technological stages: synthesis of nanomodifier by the principle of

substance formation “bottom-up” and the technology of modification of the forming solid by the principle “top-down”. Analyzing the obtained complex experimental data the optimum process parameters were established: the catalyst/cement ratio was 0.2, pyrolysis temperature (650 ± 20) °C, synthesis time 90 min, hydrocarbon gas was propane-butane mixture, catalyst Ni–MgO. The structure of the initial and synthesized samples was studied and compared on the basis of electron microscopy, Raman spectra and thermal gravimetric analysis. The materials obtained proved the presence of stable carbon nanostructures without significant inclusions of amorphous phases. Evaluation of nanomodifier influence on physical and mechanical characteristics of the construction composite has confirmed the increase of material performance up to 20 % at a dosage of nanomodified binder 1/8–1/16 of the total mass of cement.

5. Funding

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6. Conflict of interests

The authors declare no conflicts of interest.

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