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ОСОБЛИВОСТІ СТРУКТУРОУТВОРЕННЯ МОДИФІКОВАНОЇ ЦЕМЕНТНОЇ СИСТЕМИ НА РАННІХ СТАДІЯХ ТВЕРДНЕННЯ БЕТОНУ

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Анотація. Постановка проблеми. Визначення типів зв'язків, що утворюються у модифікованій цементній матриці бетону, і оцінка якості цих зв'язків у неоднорідному матеріалі для з'ясування геометричних і фізичних співвідношень між структурою модифікаторів і цементної матриці на ранній стадії структуроутворення. **Методика.** Дослідження процесів гідратації і структуроутворення звичайних і модифікованих цементних систем проводилися з використанням рентгенофазового, диференційно-термічного та мікроскопічного аналізів, інфрачервоної спектроскопії. Аналіз структурних параметрів цементної матриці бетону проводився методом ртутної порометрії, а також за адсорбцією пар води та азоту. **Результати.** Система складання з відповідною структурою формується при компонуванні і контактуванні мінеральних компонентів цементної системи і зерен заповнювачів у процесі змішування з водою, перемішування і укладання бетонної суміші. Зміна кількісних характеристик структури модифікованого бетону спостерігалася за зміни інтенсивності модифікування. Спостережувані зміни морфологічної структури цементної матриці не можуть бути випадковими, тому що вони підтверджені повторними дослідженнями за відтворюваності від 82 до 96 %. У той же час така зміна морфології новоутворень не спостерігалася у звичайному бетоні. З наведених даних очевидне значне збільшення пластинчасто-призматичної складової в цементній матриці модифікованого бетону. Зі збільшенням часу витримки кількість сформованих кристалічних новоутворень збільшується. Це можна пояснити системним підходом: морфологія структури цементної матриці - результат взаємодії систем складання і зростання. При цьому система зростання (перекристалізації і розвитку структури в часі) може ефективно розвиватися без досить повного розвитку системи складання (накопичення первинних продуктів гідратації). **Наукова новизна.** Вперше встановлено особливості структуроутворення модифікованої цементної системи, які полягають у тому, що кристали гідрату хлороксиду магнію швидко ростуть у просторі між гідратними новоутвореннями клінкерних мінералів, а механічне зчеплення, що виникає в результаті цього, зумовлює розвиток початкової міцності і жорсткості. Оскільки вільному росту кристалів перешкоджає брак простору, кристали взаємно проростають, утворюючи щільну структуру, яка зумовлює зростання міцності; вперше встановлено, що у модифікованій цементній системі, що гідратується, в результаті взаємодії різних макроіонів розвивається структуротвірний процес із переважанням активних частин, які значно перевищують його дисипативну частину порівнянно з нормальними умовами тверднення. Результати, отримані під час моделювання поведінки модифікованої цементної системи, що гідратується, показують, що в системі спостерігаються коливання концентрації проміжних продуктів гідратації, ототоженні з виникненням просторово-часової структури. **Практична значимість.** Фізико-хімічне модифікування цементної системи забезпечує зміну морфотропії кристалогідратних новоутворень, що сприятиме формуванню проектних властивостей бетону.

Ключові слова: модифікований бетон; цементна система; структурні характеристики; система зростання

ОСОБЕННОСТИ СТРУКТУРООБРАЗОВАНИЯ МОДИФИЦИРОВАННОЙ ЦЕМЕНТНОЙ СИСТЕМЫ НА РАННИХ СТАДИЯХ ТВЕРДЕНИЯ БЕТОНА

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Аннотация. Постановка проблемы. Определение типов связей, возникающих в модифицированной цементной матрице бетона, и оценка качества этих связей в неоднородном материале для определения геометрических и физических соотношений между структурой модификаторов и цементной матрицы на ранней

стадії структурообранования. **Методика.** Исследования процессов гидратации и структурообранования обычных и модифицированных цементных систем проводились с использованием рентгенофазового, дифференциально-термического и микроскопического анализов, инфракрасной спектроскопии. Анализ структурных параметров цементной матрицы бетона проводился методом ртутной порометрии, а также по адсорбции паров воды и азота. **Результаты.** Система сложения с соответствующей структурой формируется при компоновке и контактировании минеральных компонентов цементной системы и зерен заполнителей в процессе смешивания с водой, перемешивания и укладки бетонной смеси. Изменение количественных характеристик структуры модифицированного бетона наблюдалось при изменении интенсивности модифицирования. Наблюдаемые изменения морфологической структуры цементной матрицы не могут быть случайными, так как они подтверждены повторными опытами при воспроизводимости от 82 до 96 %. В то же время, такое изменение морфологии новообразований не отмечено в обычном бетоне. Из приведенных данных очевидно значительное увеличение пластинчато-призматической составляющей в цементной матрице модифицированного бетона. С увеличением времени выдержки количество сформированных кристаллических новообразований увеличивается. Это можно объяснить системным подходом: морфология структуры цементной матрицы является результатом взаимодействия систем сложения и роста. При этом система роста (перекристаллизация и развитие структуры во времени) может эффективно развиваться без достаточного полного развития системы сложения (накопления первичных продуктов гидратации). **Научная новизна.** Впервые установлены особенности структурообранования модифицированной цементной системы, заключающиеся в том, что кристаллы гидрата хлороксида магния быстро растут в пространстве между гидратными новообразованиями клинкерных минералов, а механическое сцепление, возникающее в результате этого, обуславливает развитие начальной прочности и жесткости. Так как свободному росту кристаллов препятствует недостаток пространства, кристаллы взаимно прорастают, образуя плотную структуру, которая обуславливает рост прочности; впервые установлено, что в гидратирующейся модифицированной цементной системе в результате взаимодействия различных макроионов развивается структурообразующий процесс с преобладанием активных частей, которые значительно превышают его диссипативную часть по сравнению с нормальными условиями твердения. Результаты, полученные при моделировании поведения гидратирующейся модифицированной цементной системы, показывают, что в системе наблюдаются колебания концентрации промежуточных продуктов гидратации, отождествляющиеся с возникновением пространственно-временной структуры. **Практическая значимость.** Физико-химическое модифицирование цементной системы обеспечивает изменение морфотропии кристаллогидратных новообразований, что способствует формированию проектных свойств бетона.

Ключевые слова: модифицированный бетон; цементная система; структурные характеристики; система роста

PECULIARITIES OF STRUCTURAL CHARACTERISTICS OF MODIFIED CONCRETE CEMENT SYSTEM AT THE EARLY STAGES OF HARDENING

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Abstract. Purpose. Determination of the types of bonds formed in the modified concrete cement matrix and the evaluation of the quality of these bonds in a inhomogeneous material for the determination of geometric and physical relationships between the structure of modifiers and the cement matrix at the early stage of structure formation. **Method.** Investigation of the processes of hydration and structure formation of conventional and modified cement systems was carried out using X-ray diffraction, differential-thermal and microscopic analyses, and infrared spectroscopy. The analysis of the structural parameters of the cement matrix of concrete was carried out by the method of mercury porosimetry, as well as the adsorption of water and nitrogen vapor. **Results.** The system of assembling with the corresponding structure is formed during the layout and contact of the mineral components of the cement system and aggregate grains in the process of mixing with water, stirring and placing of concrete mixture. The change in the quantitative characteristics of the structure of modified concrete was observed at the change in the intensity of modification. Observed changes in the morphological structure of the cement matrix cannot be random, as they are confirmed by repeated experiments at reproducibility from 82 to 96 %. At the same time, such a change in the morphology of new formations was not observed in normal concrete. From the given data, a significant increase in the lamellar-prismatic component in the cement matrix of modified concrete is obvious. The number of formed crystalline new formations increases with the increase of curing time. This can be explained by the system approach: the morphology of the structure of the cement matrix – the result of the interaction of systems of assembly and growth. At the same time, the growth system (recrystallization and development of the structure in time) can effectively develop

without a sufficiently complete development of the system of assembly (accumulation of primary products of hydration). **Scientific novelty.** For the first time, the peculiarities of the structured formation of the modified cement system have been established, they consist in the fact that magnesium chloride hydrate crystals grow rapidly in the space between the hydrated new formations of clinker minerals, and as the result, the mechanical bond is responsible for the development of initial strength and stiffness. Since the free growth of crystals is hindered by the lack of space, the crystals mutually germinate, forming a dense structure that causes the growth of strength; it was first established that in a modified hydrated cement system, as a result of the interaction of different macroions, a structure-forming process develops with a predominance of active parts that significantly exceed its dissipative part in comparison with normal cure conditions. The results obtained in modeling the behavior of the modified cement system, which is hydrated, show that the system has fluctuations in the concentration of intermediate products of hydration, identified with the appearance of the spatial-temporal structure. **Practical relevance.** Physico-chemical modification of the cement system provides a change in the morphotropy of new crystalhydrate formations, which will contribute to the formation of the design properties of concrete.

Keywords: *modified concrete; cement system; structural characteristics; growth system*

Introduction. Prospects for the further development of high-performance concrete production and scientific and technological progress are largely determined by the knowledge of the laws and methods of controlling the processes of the structural formation of the cement matrix of concrete, taking into account the required high level of product quality and the criteria of the resource intensity of production [2].

In this connection, first of all, it is important to analyze the general patterns of formation of the structure of the material in the complex process of physical and chemical modification of the concrete mixture for monolithic structures.

Purpose. Determination of the types of bonds formed in the modified concrete cement matrix and the evaluation of the quality of these bonds in a inhomogeneous material for the determination of geometric and physical relationships between the structure of modifiers and the cement matrix at the early stage of structure formation.

Method. Investigation of the processes of hydration and structure formation of conventional and modified cement systems was carried out using X-ray diffraction, differential-thermal and microscopic analyzes, and infrared spectroscopy. The analysis of the structural parameters of the cement matrix of concrete was carried out by the method of mercury porosimetry, as well as the adsorption of water and nitrogen vapor.

Results. From the general positions, the structure formation of the modified cement matrix of concrete can be presented as a transformation of the system of assembly of the

initial mineral components and the system of growth of new substances that emerge at different stages of the technology [3].

The system of assembling with the corresponding structure is formed during the layout and contact of the mineral components of the cement system and aggregate grains in the process of mixing with water, stirring and placing of concrete mixture. The system of assembly is formed from the finished structural elements in the continuous occurrence of new elements of the structure, as well as intergranular pores and interparticle contacts [1; 4].

An analysis of the formation of assembly systems and their parameters was made by the mechanics of granular media. The content of this analysis is determined by the requirements of obtaining data on the patterns and nature of packaging particles in connection with their granulometric composition. In the technological sense, this is expressed in the form of tasks for selecting the optimal granulometry of the components, the optimal ratio of their volumes on the criterion of the density of the package, the minimum water content of the mixture.

At the grinding stage of the original components, the task of analyzing the assembly system can be to determine the degree of agglomeration of particles, the homogeneity of the distribution of components in each microobject of the product when grinding [6; 7].

The task of the homogeneity of the mixture is characteristic both for the stage of preparation of the concrete mixture, and, especially, when it is modified and placed.

At physical modification of the concrete mixture, the content of the problem involves studying the phenomena of repacking particles in connection with the application of physical influences. When solving these problems there's simultaneously preparing the optimal conditions for the formation and development of the system of growth (structure formation).

The system of growth with its inherent structure is created as a result of a combination of various by mineralogy, morphology and the size of dispersed particles of new formations that emerge in the process of hydration, accompanied by a physical and chemical modification of the cement matrix. In this case, the growth system is created from new formations during the technological process, and this is its fundamental difference from the system of assembly [5].

The development of the growth system reflects, first of all, the process of accumulation of cementing agent with its qualitative changes occurring in conditions of physical modification. The basis of the analysis of the formation and parameters of the growth system is the physico-chemistry of the heterogeneous processes of the emergence of a new disperse phase. The content of this analysis is conditioned by the need to obtain information on the kinetics and the dynamics of the formation of hydrated compounds, their components, the degree of dispersion, the peculiarities of contacting in the crystal intergrowth, as well as the evolution of the pore space of the cementing agent during physical modification. In practical terms, this information, which correlates with the modes of technological processes, is necessary for their management in accordance with the purpose of obtaining concrete with the necessary high quality [8; 10].

The growth system develops on the basis and within the system of assembly, transforming it, interacting with it and being under the influence of physical and chemical modification of the binder. This is expressed in the fact that the system of growth of modification with the accumulation of cementing agent takes up part of the disappearing volume as a result of hydration, as

well as a sharp narrowing of the intergranular space of the initial assembly system. The result is the following. First, the initial value of the intergranular cavernosity of the assembly system is not preserved in the synthesis of new formations. The cavernosity of the original system of assembly (without considering the growth system) usually does not increase, but under the action of the compacting is sharply reduced. In this case, the volume of the cementing agent required for grouting the aggregate grains, appears to be greater than the volume of voids formed in it. A new volume of intergranular space is almost completely filled with the cementing material with volume and pore size distribution specific for it. Thus, the structure of the porosity of modified concrete is fundamentally modified.

The interaction of the assembly and growth systems lies in the fact that during the formation of a cementing agent its contact with the surface of aggregate grains and other source components is formed. The influence of the system of assembly on the growth system at the same time is expressed by the fact that the kinetics of the accumulation and crystallization of the cementing agent, the dynamics and the mechanism of formation of the contact zone depend on the crystallographic characteristics of the values of the surface area, the specific surface energy of the particles of the assembly system, finally, on the parameters of its porous space. As experimentally proved, we can assume that the structure of the new material is a composition of the systems of assembly and growth at the appropriate stage of their interaction [9].

The resulting structure depends on the combination of all structure-forming factors that can be characterized by a certain potential of structure formation or the boundary of the formation of the cementing matrix. The magnitude of the potential is a quantitative expression of the degree of completeness of this structure in relation to the limit, ideal structure. Incomplete realization of this potential in the obtained material will be characterized by the appropriate degree of incompleteness of the structure formation process, which determines

the level of material quality according to its construction and technological properties.

Quantitatively, the degree of completeness of structure formation can be estimated by the ratio of parameters of the state of the structure, fixed at *i*-th technological conditions, to the boundary state with the maximum possible density of new formations and the limiting specific activity of surface bonds, which is principally achievable with the new technology. The degree of completion of the structure formation of *Sc* for the entire range of technological conditions is normalized in the range of values from 0 to 1.

Our quantitative estimates of the structure of various materials are implemented with some controlled reliability. For verification of structural investigations of various concrete the method of selection and preparation of statistically representative samples of the cement matrix is substantiated and applied. Special studies have established that the statistical reproducibility of the characteristics of the composition, parameters of structure, indicators of the state of the material corresponds to errors that do not exceed 7...9 %, and estimates of physical and mechanical properties - up to 9...14 %. The results of the studies are presented in the Table 1 and on Figure 1.

With almost constant ratio of volumes of hydrated phases and porous space, they undergo regular qualitative changes in time. In the solid phase, as the cementing agent exponentially accumulates, the volume of fossil minerals also exponentially decreases. Although the cementing agent at various stages of its formation is represented by a set of highly basic forms of hydrated new formation, their structure depends on the technological conditions of the formation.

The dynamics of the morphological composition of the cementing agent of

modified concrete is determined by the continuous decrease of the relative volume fraction of the sub-microcrystalline component during the entire period of curing; growth of the part of short-fiber, decrease of the lamellar-prismatic component in the stage of accumulation of new formations; a decrease in the proportion of short-fiber and an increase in the proportion of the lamellar-prismatic component at the stage of recrystallization of new formations with the accumulated (almost unchanged) amounts of the latter.

The change in the quantitative content of the cementing agent while simultaneously developing the process of morphological transformation, crystallization and recrystallization of synthesis products determine the ambiguous nature of the change in the magnitude of their total specific surface area. Specific surface passes through its maximum value in the first hours of synthesis of new formations and subsequently significantly reduced.

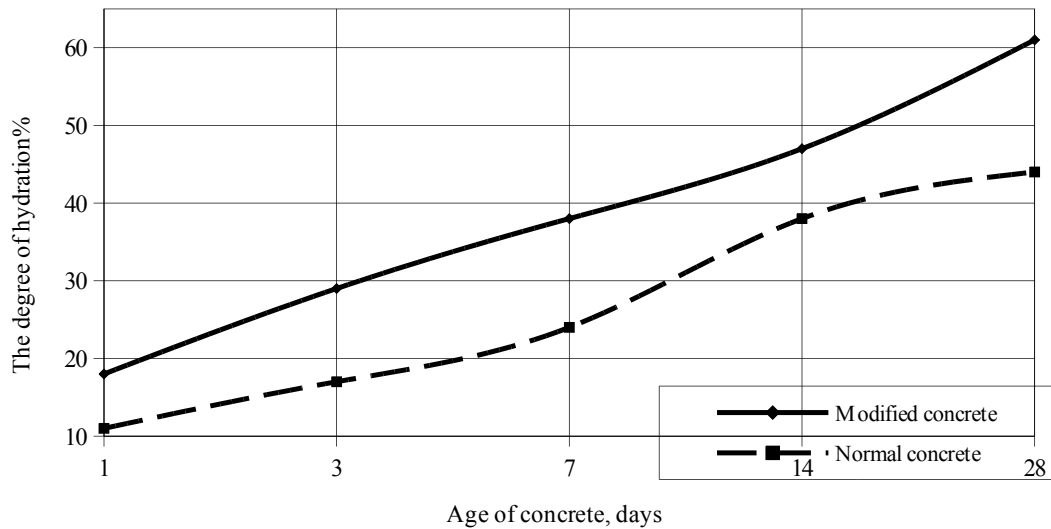
The structure of porosity at a practically constant value of porosity of a material is determined by the change in the distribution of micropores in size as the overgrown grain void of the system of assembling the initial particles with the cementing agent and the simultaneous change in the mineralogical and morphological composition, the size of the crystals. The effective pore radius as a measure of the estimation of the function of their distribution in size may decrease in the period of accumulation of the cementing agent in 4...5 times, and in the period of recrystallization of new formations, the consolidation of the crystals in the growth again increase in 1.5...2 times compared with its minimum value.

Table 1

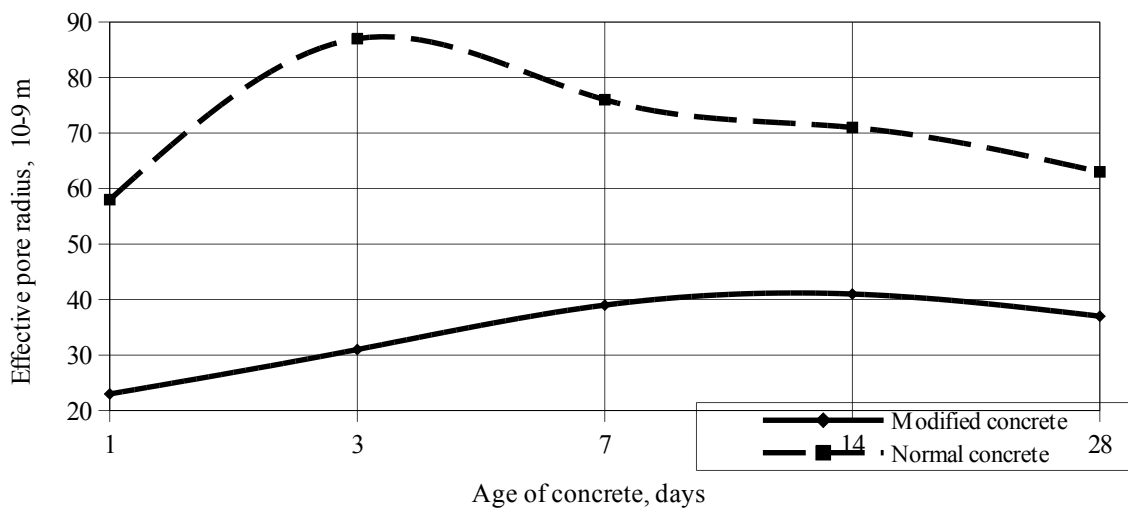
Change of the morphological composition of the cement matrix during the curing process

Type of concrete	Type of structure	Volume of new formations, cm ³ /cm ³ of cement matrix at age, days				
		1	3	7	14	28
Modified	Submicrocrystal	0.42	0.39	0.24	0.28	0.25
	Short-fiber	0.52	0.57	0.59	0.43	0.48

	Lamellar-prismatic	0.16	0.04	0.17	0.29	0.27
Normal	Submicrocrystal	0.22	0.29	0.34	0.31	0.28
	Short-fiber	0.42	0.66	0.63	0.63	0.58
	Lamellar-prismatic	0.36	0.05	0.03	0.05	0.14



a – the degree of hydration of the concrete cement matrix



b – change in the porosity of the cement matrix of concrete

Fig. 1. Structural characteristics of various types of concrete

The formation of crystalline intergrowth is due to the formation of contacts of adjoining, merging and germination of individual crystals and druses. During hardening there's a developing process of transition of the contacts from the adjoining contacts to the joints, but with a tendency to increase the degree of damage, defects of individual crystals. The bonding zones of the cementing agent with the surface of the aggregate grains are formed as an adjoining contact.

The results of experimental studies of the structure of the cement matrix are shown in Table 2, they show the consistent patterns for modified concrete. When changing the technological conditions of compacting or squeezing out water, quantitative expressions may be different, but their general character is preserved.

The change in the quantitative characteristics of the structure of modified concrete was observed at the change in the

intensity of modification. Observed changes in the morphological structure of the cement matrix cannot be random, as they are confirmed by repeated experiments at reproducibility from 82 to 96 %.

At the same time, such a change in the morphology of new formations was not

observed in normal concrete. From the given data, there's obviously a significant increase in the lamellar-prismatic component in the cement matrix of modified concrete. With increasing time of exposure, the number of formed crystalline new formations increases.

Table 2

Morphological structure of cement matrix of modified concrete

Morphological structure	Submicrocrystalline	Short-fiber	Lamellar-prismatic
Mineralogical composition	High-basic calcium hydrosilicates CHS	mainly CHS of tobermorite group	tobermorite with high processing time
Number of contacts per unit volume	10^7	10^5	10^4
Type of contacts of particles and crystals	Contacts of adjoining of globular particles	Combination of contacts adjoining and merging of crystals	Combination of adjoining, merging and germination of crystals
Size of particles and crystals, medium-sized section/ medium-length, 10^{-6} m	0.12/0.17	(0.14/1.1)... (0.45/4.7) <u>0,10</u> <u>0,5</u> 1,0 ... 5,0	(0.75/2.2)... (1.15/5.3) <u>0,80</u> <u>1,3</u> 2,4 ... 5,0
The average effective pore radius, 10^{-9} m	6...9	22...37	42...75
Specific surface area, m^2/g	470	120...210	43...54

This can be explained by the system approach – the morphology of the structure of the cement matrix – the result of the interaction of systems of assembly and growth. At the same time, the growth system (recrystallization and development of the structure in time) can effectively develop without a sufficiently complete development of the system of assembly (accumulation of primary products of hydration). The developed system of assembly hydration of cement particles in favorable conditions of the hydraulic pressure of the surrounding water due to thixotropy will improve the morphology of structural new formations. However, this phenomenon is not observed in normal concrete, where there is no modification of the structure. According to experimental data obtained by complex methods of studying the structure, development in volumes of similar morphological structures can reach 20 %. At the same time, the degree of hydration increases by 11...14 %.

Scientific novelty. For the first time, the peculiarities of the structured formation of the modified cement system have been established, they consist in the fact that magnesium chloride hydrate crystals grow rapidly in the space

between the hydrated new formations of clinker minerals, and the mechanical bond resulting from this is responsible for the development of initial strength and stiffness. Since the free growth of crystals is hindered by the lack of space, the crystals mutually germinate, forming a dense structure that causes the growth of strength.

It was first established that in a modified hydrated cement system, as a result of the interaction of different macroions, develops a structure-forming process with a predominance of active parts that significantly exceeds its dissipative part in comparison with normal cure conditions. The results obtained in modeling the behavior of the modified cement system, which is hydrated, show that the system exhibits fluctuations in the concentration of intermediate products of hydration, identified with the appearance of the spatial-temporal structure.

Practical significance. Physico-chemical modification of the cement system provides a change in the morphotropy of crystalhydrate new formations, which will contribute to the formation of the design properties of concrete.

Conclusions. It was established that the process of structure formation of the modified

cement system in the presence of organo-mineral modifying complex leads to a change in morphology of new formations in comparison with usual concrete with quantitative changes in the ratio of volumes of cryptocrystalline, needle-fibrous and lamellar-prismatic components of concrete cement matrix.

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