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Al-Qadisiyah Journal for Engineering Sciences

Journal homepage: http://qu.edu.iq/journaleng/index.php/JQES



Use of Bypass Cement Dust from Al-Kufa Cement Factory for Production of Glass Ceramic Material

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ARTICLE INFO

Article history: Received 15 July 2019 Received in revised form 4 August 2019 Accepted 8 August 2019 Keywords: Bypass Glass ceramic Main material Reinforcing materials Sintering process

ABSTRACT

Bypass cement dust was supplied from the Alkufa cement factory, which has been used in preparing glass ceramic material. The percentage of bypass cement dust, which is included in the overall batch, is about 30 wt %. The bypass cement dust composition has been modified by adding other materials like marble, granite, and silica, to prepare glass ceramic material by powder technology. The prepared samples with different ratios ingredients were pressed under a load of 15 tons/cm² for 90 sec, in a steel mold of 30 mm in diameter, liquid phase sintering is carried out by using High-Temperature Furnace at 100 - 1250 ° C and 208/240 VAC. Techniques including SEM (Scanning electron microscopy) and XRD (X-ray diffraction) tests were then used to evaluate the produced material. The obtained glass ceramic material has high bulk and apparent density, low apparent porosity, high compression strength and hardness , low electrical and thermal conductivity. The XRD and SEM results showed that glass ceramic materials are formed. The phase transformation has happened and the titanite, wollastonite, diopside, periclase, and silica condense clusters phase are formed, which are the important phases of glass ceramic. Tests show that the sample A5 contains 38% bypass cement dust, 30% granite, and 32% silica; and the sample C5 contains 28% bypass, granite 30%, marble 10%, and silica 32%; and the sample B4 contains 59% bypass, 25% marble, and 16% silica. Showing properties A5 and C5 which conform to that of glass ceramic properties better than B4. The research is considered a pioneer work in the field of investment of dust of bypass cement for producing glass ceramic materials. It represents an important step and could be the basis for future researches leading to the achievement of glass ceramic production in Iraq.

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1. Introduction

Glass ceramic is considered to be one of the most versatile and precise materials which are used in different fields of industry. Glass ceramic has a wide range of considerable properties such as heat insulation, high strength, thermal shock resistance, high- temperature stability, toughness, and capabilities of isolation [1]. Glass ceramic materials, which have been prepared via controlling glass crystallization can be used for different purposes depending on their uniform, reproducible fine grained microstructure, the absence of porosity and many other different characteristics that may be regulated by changes in composition and the process of heat treatment. In fact, glass ceramics that are based on the structures of chain silicate were established [2]. A number of works were performed on the diopside- anorthite- celsian (MgCaSi₂O₆- CaAl₂Si₂O₈-BaAl₂Si₂O₈) system [3]. The nature and characteristics of crystalline phases and the material microstructure were reported as the most significant parameters that affect technical characteristics of glass ceramic [4]. Controlled bulk crystallization has been produced by the

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https://doi.org/10.30772/qjes.v12i2.604 2411-7773/© 2019 University of Al-Qadisiyah. All rights reserved. devitrification technique which has been most utilized used to produce glass ceramics. Researchers have been mostly focused on glass in systems like SiO₂-Al₂O₃, MgO-Al₂O₃-SiO₂, and CaO-Al₂O₃-SiO₂ [5]. In order to avoid uncontrolled crystallizing, controlled surface crystallizing has been performed on glasses in systems MgO-Al₂O₃-SiO₂ and CaO-Al₂O₃-SiO₂ [6]. The basic phases of crystalline have been found to be anorthite (CaAl₂Si₂O₈), titanite (CaTiSiO₅), wollastonite (CaSiO₃), spinel (MgAl₂O₄) forsterite (Mg₂SiO₄), and cordierite ((Mg, Fe)₂Al₄Si₅O₁₈), [6]. Research on CaO-MgO-Al₂O₃-SiO₂ system is significant as well in order to understand the reaction that occurs in the rocks, MgO refractories, and blast furnace slag [7]. The glass ceramic material does not have a fixed chemical composition or a specific crystal structure, which is an oxide. It is composed of the matrix component material and the added material for reinforcement and the auxiliary in the sintering, and there are other materials that can be added for installation to improve the mechanical properties and to control on the color of the product [7]. Glass ceramics are composite materials that generally consist of combining two or more materials to obtain a material with geometric and physical properties that differ from the properties of the composition and the resulting material is better than its components. Composite materials consist of two phases matrix phase and reinforcing phase[8]. In the scientific literature, ceramic materials are compounds between mineral and non-metallic elements, most of which are oxides, nitrides, and carbides. It has a complex crystalline structure with ionic, covalent, or common bonds [9]. Cement kiln dust is produced in cement kilns and related devices. Dust from raw mixture and plant surroundings are typically contained by water sprays and closed systems. Throughout burning, gas flows entrain a considerable amount of dust which makes up a portion of the exit gas from the kiln. The dust of the cement kiln which has been generated is strongly dependent on the chemistry of the raw materials, velocities of the gas in the kiln, and type of the process. Other elements like the efficiency of the kiln and dust collection system design are vitally important. A variety of approaches have been utilized for capturing dust, which includes bag filtration and electrostatic precipitation. In the case where the dust of the cement kiln includes low amounts of chemicals (less than or equal to 75wt%) like the chlorides and alkalis, then there is a possibility of returning dust in the kiln as a part of raw mixture [10]. Dust of cement kiln which has not been reused this way may be utilized in many other different industrial applications, for instance, is a form of subbase in the construction of road giving its hydrating features, in a form of a cement additive in the process of grinding, as a backfill material in mining, and as an alkali source in chemical processing (such as Potassium sulfate for extracting potassium oxide) [11]. Cement dust is an undesirable cement industry by-product, instead of being discharged in the atmosphere, which is why, it is typically kept behind filters, as a sufficient, active particulate residue. The concentration of cement dust in the chimneys does not exceed 150 g /cm3 at the outer boundary of the Al-Kufa cement factory in the direction of the wind [10]. Efforts to recycle the dust result in a maximal utilization of only around 10% to 20%, the rest amount which is quite large is, in general, landfilled or loaded on trucks and thrown in near open surface impoundments, the dust of disposal permits massive amounts of light-weight, erosive, fine powder to be released to the environment with the wind blowing, and that poses severe health and environmental risks. The chemical composition of bypass cement dust indicates the potential of using it in glass ceramic and glass industries [11]. Due to the fact both those industries are known for having numerous applications in commerce, for instance, in the microelectronics industry and constructions, utilizing bypass dust of cement for the production of glass ceramic materials is greatly significant in technological scientific, and economic aspects [11]. In this research, bypass cement dust from Al-Kufa cement factory is used to produce glass ceramic materials.

2. Materials and Methods

2.1. Materials used

The matrix material powder (bypass cement dust) was supplied from the Al-Kufa cement factory. The reinforcing materials (granite, marble, and silica) were obtained from the Iraqi market.

2.2. Experimental procedures

The first stage did the chemical composition of matrix material (bypass cement dust) and reinforcing materials (granite and marble) is presented in Table 1. XRF was used to analysis the reinforcement material of silica, showing the purity of 62.4% .In the second stage, the reinforcement materials were broken by jaw crusher (42781 Haan-2013) . The material size was reduced by a ball mill (Retsch, Model PM 100) at speed 300 rpm for 25 min. Then the materials were sieved by sieve shaker (AD60-01) according to ASTM at vibration 60 Hz for 15 min to obtain particle size ≤ 0.25 mm. The third stage mixing of the powders by a ceramic container perform by different ratios of matrix material (bypass cement dust) and reinforcing materials for 50 min, as shown in Table 2. The fourth stage did the pressing process of the specimens were compression by using load 15 ton for 90 sec and using a cold pressing technique in one direction (Uniaxial pressing) in a steel mold with diameter 30mm. The last stage did the sintering process by using an electric furnace (German Nabertherm). The samples were sintered at a temperature of 850 °C for 8 hrs for heating rate of 2°C/min and soaking time of 120 min. Fig. 1. shows the sintering temperatures against the time for sintering. It is observed that the almost densification occurs at a sintering temperature of 850°C.



Figure 1. Heat treatment cycle in the sintering process.

2.3. Physical and Mechanical Testing

- The samples were examined by the following devices:-
- 1- X-ray diffraction analysis was carried out at the Geology Department, College of Sciences, University of Baghdad. The XRD analysis was performed at a wavelength of 1.54060 (A) using the Cu Kά X-ray source and 2θ range from 30° to 90° with voltage 40.0 (KV). XRD was used to determine the phases of the samples and their types (BRUKER) and scan rate (speed) 5-10° min.
- 2- SEM and EDX analysis Electron microscopy scanner (T scan) has been used to study the structure of microparticles by using scanning electron image type (VEGA3 LUM) and Energy dispersive X-ray spectroscopy (EDS) type (Oxford Max3) to obtain a primary characterization of the materials. The analysis was performed at the University of Technology / Department of Production Engineering and Metallurgy.

- 3- According to Archimedes rule, Bulk Density, Apparent Porosity & Water Absorption were tested using standard (ASTM C373-88) and a sensitive electrical balance (Sartorius) with an accuracy of 5 digits.
- 4- Compressive strength To perform the test (Microcomputer Controlled Electric Universal Testing Machine was used to evaluate the compression strength of the prepared materials by returning to standard ASTM E9-89. The machine has the capacity that does not exceed 180 KN.
- 5- Electrical and Thermal conductivity Electrical and Thermal conductivity of samples was examined at the University of Baghdad, College of Education Ibn-Alhaytham for sciences of drainage in the service laboratory.
- 6- Hardness samples were measured using the micro Vickers method by a device (Digital Micro Hardness Tester). According to the ASTM E-384-11, and using a load of 1000 g for 10 sec.

Table 1. Chemical composition of matrix material (bypass cement dust) and reinforcement materials

Materials	SiO %	Fe ₀ %	Al ₀ %	TiO_%	CaO%	MgO%	SO %	LOI%	Na O%	K ₀ %	CL%	Total%
Bypass cement dust	13.14	1.49	3.70	0.14	43.00	3.90	2.900	27.90	0.87	1.02	0.99	99.05
Marble	0.27	0.09	0.17	0.01	54.17	1.28	0.010	43.90	0.04	0.01	<0.02	99.96
Granite	76.18	3.24	6.84	0.30	1.00	0.18	0.020	0.68	4.09	7.41	0.02	99.96

Table 2. Percentage of matrix material (bypass) and reinforcing materials

Sample	Bypass wt%	Granit wt %	Marble wt%	Silica wt%
A5	38%	30%	0	32%
B4	59%	0	25%	16%
C5	28%	30%	10%	32%

3. Results and discussion

3.1. XRD Analysis

Fig. 2. shows the formation of crystalline phases for the sample A5 after thermal treatment. The titanite is formed due to the combination of oxides (TiO, SiO₂, CaO), which leads to complex composite (CaTiSiO₅) (31.9%), causing an improvement in the hardness [9]. The formation of wollastonite (CaSiO₃) (19.9%), is produced by the combination of CaO with SiO2 which improves the performance parameters due to its fluxing properties, freedom from volatile constituents, whiteness, and the acicular particle shape and decreases shrinkage. Also, the formation of diopside MgCaSi₂O₆ (16.9%) is formed as one of the main crystalline phases in glass ceramic. The formed compound Si₂O₄ (29.4%) is a condensed phase, which consists of clusters of extremely small particles, these particles are covalently bound, which is responsible for decreasing thermal and electrical conductivity, due to covering the sample by mantle. Periclase MgO (1.7%) is an important material generally used in refractories manufacturing because of its low thermal expansion coefficient, excellent chemical stability, and resistance to thermal shock. Increasing the crystal form of Periclase helps to remove any holes, porosities or any cracks formed due to shrinkage and other thermal expansion [9].

Fig. 3. shows the formation of crystalline phases of Pyrochlore $(Ca_2Ti_2O_6)$ (42.1%) on the sample B4 after thermal treatment, which improves the thermal insulation property of the material.

The formation of Anorthoclase ((Na,K) (AlSi₃O₈) (19.5%), which is a crystalline solid composite improves the hardness of material [11]. The Diopside MgCaSi₂O₆ (17.5%) is formed as well, which is one of the main crystalline phases of producing glass ceramic. The properties of sample B4 is better than sample A5 because of the high percentage of Diopside in B4. The percentage of Periclase (10.4%) in B4 is greater than its value in A5.



Figure 2. XRD pattern of A5 sample



Figure 3. XRD pattern of the B4 sample.

Fig. 4 shows the formation of crystalline phases for the sample C5 after thermal treatment. These phases are wollansite (38.3%), quartz (29.6%), titanite (24.0%), calcium hexaaluminate (3.6%) cristobalite (3.4%) are formed except diopside and periclase . However, Diopside is one of the main crystalline phases in glass ceramics [12] beside Periclase in spite of its low percentage, so that sample A5 represents glass ceramic material in large extent as a compared to the sample C5.

In addition to that C5 sample contains a high percentage of SiO_2 and cristoballite which is a high-temperature polymorph of silica, meaning that it has the same chemical formula as quartz, SiO_2 , but a distinct crystal structure. Both quartz and cristobalite are polymorphs with all the members of the quartz group [12].



Figure 4. XRD pattern of C5 sample

3.2. SEM & EDS Analysis

Figs. (5,7 and 9) show the SEM images of the prepared samples. The crystallization starts from the external surface toward the inside region. In A5 sample Diopside crystal began to grow as thin needles from the surface of the glass ceramic specimen towards the centre. It shows also

the formation of the needle like structure, showing skeletal herringbone structure which represents Diopside [11].

In the C5 sample, the bladed circular Wollastonite is changed to the texture in glass ceramic sample from rough (coarse) to soft (fine) particles which may attribute to increasing in viscosity due to increase in the content of SiO₂% and CaO%.

The results show that the presence of MgO and CaTiSiO₅ in glass ceramic will increase the tendency of bulk crystallization of glass ceramic. Ca⁺² ions likewise are responsible for bulk crystallization in both A5 and C5 samples. The result of a possible change in the coordination state of Ca⁺² ions in silicate glass ceramic has been subject to considerable debate. The result of a possible change in its coordination state is from cubic to octahedral structure. The coordination states depend, to some extent, on alkalinity or basicity of silicate. The Ca⁺² ions would, therefore, exist in both six-fold coordination (octahedral) in wallastonite and eight-fold coordination (cubic) in titanite in sample A5. In sample C5 both eight-fold coordination (cubic) in wollastonite and six-fold coordination (octahedral) in titanite are observed in glass ceramic structure because of increasing the percentage of phases, due to increasing the chemical coordination in silicate glass ceramic material [11-4].

The crystallization in sample B4 is begun by surface and bulk nucleation. Crystallization occurs through the entire volume of sample B4. This is desirable for enhancing the crystallization of glass ceramic through bulk nucleation, which tends to form crystalline glass ceramic products.

Figs. (6,8 and 10) show the EDS results of glass ceramic of sample A5,C5, and B4. It is observed that the A5 sample consists of Mg, because the sample contains Diopside and Periclase (MgO), while the sample C5 does not have Mg. Moreover, Fe and S elements are found in A5 sample, while the sample C5 is free from these elements. The amount of Fe in sample A5 is greater than its content in sample C5 due to the formation of Fe₂O₃. The Mg is included in sample B4, because of the structure of the sample which consists of Diopside and Periclase. Further, Ca, Ti, Na, K, Al are involved in the structure due to Pyrochlore, Mullite, and Sillimanite phases.



Figure 5. SEM of A5





Figure 6. EDS of A5





Figure 8. EDS of C5





Figure 7. SEM of C5

Figure 9. SEM of B4





3.3. Bulk Density

Fig. 11 shows the variation in bulk density of the sintered materials with the change in the main and the reinforcing contents glass ceramic . It is indicated that the decrease in the percentage of bypass cement dust has led to an increase in bulk density. The density varies with the range (4.57, 4.84, 4.976) g/cm³. The bulk density in A5 sample was increased with increasing the sum percentage of reinforcement material. Although, Marble was not found in this sample. The increase in density is due to the high calcium oxide content, which is about 43 % of the cement dust, and has a density of 3.35 g/cm³, also SiO₂ which formed 13.14 % of cement dust for a density of 2.641 g/cm3. Furthermore, the reinforcing materials have high density values which are 3.125 g/cm3 and 2.641 g/cm³ for the granite and silica respectively, which may contribute to an increase in density of A5 sample. It is noticed that the bulk density of B4 sample is less than A5 because of absence granite due to increasing the bulk density in B4 in the range (3.766, 3.821, 4.117) g/cm³. It is noticed that the bulk density in sample C5 is greater than the density in B4, while it is less than the density in A5. It is noticed that the bulk density reached maximum value when there is no addition of marble to A5 sample and value of C5 which gives better results than B4 samples. The high density of the samples A5 and C5 are due to the high density of the granite. Hence the porosity is not taken into account. As a result,

bulk density is lower than, or equal to (in the absence of porosity), true density. This is complying with study in [12], which explains the reason for increasing the density of sample A5 due to the presence of cement dust and granite at a high percentage. In addition, the sintering of the compress reduced the size of the spaces between particles. This conclusion does not agree with [13] where the compositions due to powder metallurgy where the density decreases with the decreases of the reinforcement materials because increasing porosity.



Figure 11. Relationship between the bulk density and the percentages of main & reinforcing particle contents after the sintering process.

3.4. Apparent porosity

Fig. 12 shows the relationship between the percentages of the main contents of glass ceramic and apparent porosity after the sintering process. It is noticed that the decrease in the percentage of the cement dust has led to decrease in the porosity within the range (0.740, 0.731, 0.656)% in sample A5 when the percentage of bypass cement dust 38wt%. The porosity in sample B4 is decreased within the range (0.983, 0.952, 0.832) % with increasing the percentage of bypass cement dust to 59 wt%. The porosity in sample C5 is varied as following (0.763, 0.820, 0.857%) which is less than the porosity values in sample B4. However, the porosity in sample C5 is greater than porosity values in sample A5.

Fig. 12 also shows the variation in apparent porosity with the reinforcing materials (granite, marble, and silica) after the sintering process. The percentages of reinforcement are 41%, 62% and 72% for samples B4, A5, and C5 respectively. It is noticed that the increase in the percentages of the reinforcing materials has led to decrease in the porosity within the same range. On the other hand, if the quantity of the reinforced material reduced, the apparent porosity will increase . It is noticed that the samples A5 and C5 have fewer porosities, although they have 62 wt% and 72 wt% percent of the reinforcement materials in the absence of marble in A5 because the high density for the crystallization of the phase formed in A5 and C5. In general, when the density increased the porosity will decrease. Also, the crystallization occurred due to sintering will reduce the porosity because the lowering of vacancies between crystals which leads to improving microstructure of the material developed [14]. For sample B4, the porosity is increased because of the decrease in density due to the absence of granite which has the highest density.



Figure 12. Relationship between the apparent porosity and the percentage of main & reinforcing particles contents after the sintering process

3.5. Water Absorption

Fig. 13 shows the relationship between the percentages of the has and reinforcing contents of glass ceramic and water absorption after the sintering process. It is seen that the decrease in the percentage of bypass cement dust with increasing in the percentages of reinforcing material to 62 wt% in absence marble has led to decrease in water absorption ability in sample A5 within the range (0.310, 0.218, 0.212)%. It is detected that the increase in the percentage of bypass cement dust and decreasing in the percentages of reinforcing material to 41wt% in absence granite has led to decrease in water absorption ability in sample B4 within the range (0.425, 0.423, 0.421)%. It is noticed that the decrease in the percentage of bypass cement dust will increase in the sum of percentages of reinforcing material to 72 wt% led 56 wt% to reduce water absorption ability in sample C5 within the range (0.312, 0.230, 0.221)% which is less than water absorption in B4 and it is greater than water absorption in A5. The reason is due to the same reasons mentioned in the apparent porosity where the high temperature of the gradual sintering which leads to evaporation of the substance PVA which is used in the compression process. It is noticed that the water absorption of samples A5 and C5 are less than sample B4, This is can be rationalised to the increase in the amount of reinforcement, which reduces the free spaces in material, leading to lower water absorption [14-15].



Figure 13. Relationship between the water absorption and the percentages of main & reinforcing particle contents after the sintering process.

3.6. Hardness

Fig. 14 shows Vickers's hardness in samples C5 and A5 greater than hardness of B4.The reason for the increased hardness in A5 and C5 is the phases that were formed after sintering process and indicated in the described previously. XRD and These phases (Diopside MgCaSi₂O₆),(wollastonite CaSiO₃) ,(Periclase MgO), (Titanite CaTiSiO₅), (Quartz SiO₂),(Cristobalite SiO₂). Because the sample A5 contains diopside and titanite which have high hardness and also the sample C5 contains wollastonite ,Cristobalite, Quartz, and titanite, which have high hardness. It noticed the hardness of C5 a little more than A5 because absence the marble from the A5 led to reducing hardness. Although B4 has good hardness, less than A5 & C5 samples. So that samples A5 and C5 comply with the properties of glass ceramic [14-16]. These phases were formed during the sintering process, causing very strong coherence between the particles to produce a rigid body with high Vickers hardness.





3.7. Compressive Strength

Fig. 15 shows the change in compressive strength with the content for reinforcement. The compressive strength in C5 is greater than B4 and A5 because the C5 sample contains high percentage of reinforcing material, which has worked as a barrier to the growth and progress of the cracks, and thus gives a good resistance to distortions which tends to the production of coherent particles within the samples of high compressive strength. On the other hand, the high sintering temperature tends to increase the adhering between the particles and minimize the pores and void space between them which is also increased the compressive strength [14].



Figure 15. Relationship between the compressive strength and the percentages of main & reinforcing particles content after the sintering

3.8. Electrical Conductivity

Fig. 16 shows the changes in electrical conductivity with the content of reinforcement. The electrical conductivity of B4 is greater than the electrical conductivity of sample A5, because of the presence of Pyrochlore ($Ca_2Ti_2O_6$) phase (42.1%) in B4 which has low electrical conductivity. It is observed that the electrical conductivity of C5 is greater than electrical conductivity of sample A5. However, it is less than the conductivity of sample B4.

A5 sample is better than C5 and B4 because A5 contains Si_2O_4 which form condensed phases called clusters consisting of exceedingly small particles that are covalently bound and responsible for decreasing thermal and electrical conductivity, due to the formation of mantle covered the sample. The decrease in electrical conductivity may be attributed to an increase in the interfacial area due to the decrease in the average grain size [17]. Another reason for the decreasing in electrical conductivity is due to ionic – covalent bonding which does not form free electrons. The Covalence resulted from the sharing of the two electrons in the outer shell of Ca and Mg atoms which are found in wollastonite and diopside, i.e. formation of immobile divalent alkali earth ions which block and occupies the interstitial sites and thus reduce the mobility of the alkali ions. The electrical conductivity of glass ceramic highly depends on temperature. At room temperature, the mobility of ions is very small, while it increases with raising temperature [17].



Electrical Conductivity

Figure 16. Relationship between the electrical conductivity and

percentages of main & reinforcing particles content after the sintering process.

3.9. Thermal Conductivity

Fig. 17 shows the variation in thermal conductivity with the content of reinforcement. The thermal conductivity of samples B4 and C5 are greater than the thermal conductivity of sample A5. This is attributed to including Si_2O_4 in sample A5. This helps to form condensed phases called clusters consisting of exceedingly small particles which are covalently bound and responsible for decreasing thermal and electrical conductivity. Further, a layer from Mantle may form on the sample, causing decrease in conductivity.

Heat transport in dielectric solids like glass ceramic depends on the elastic vibration of the lattice, which has a limited effect due to internal boundary scattering. The addition of bypass cement dust and reinforcing materials increase crystal complexity which tends to increase the limitation of the effect of boundary scattering and lower the heat transport, that means low thermal conductivity [18].

Thermal Conductivity



Figure 17. Relationship between the thermal conductivity and percentages main & reinforcing particles content after the sintering process

4. Conclusions

- 1- Compressing powder of the samples under a load of 15 ton/cm² is ideal to produce appropriate samples for testing.
- 2- The sintering process occurred at 850 °C for 8 hours produced appropriate samples that have suitable properties and undergoes phases formation.
- 3- Increase the bulk and apparent density, hardness, compressive strength and reduce the porosity, water absorption, electrical and thermal, therefore, it is noticed the samples A5 and C5 better than B4.
- 4- There is a possibility to use bypass cement dust from Al-Kufa Cement Factory for the production of glass ceramic.

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