FTD-TT- 63-275

# TRANSLATION

## TRANSACTIONS OF THE INSTITUTE OF CONSTRUCTION AND CONSTRUCTION MATERIALS

(SELECTED ARTICLES)

FOREIGN TECHNOLOGY
DIVISION

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# UNEDITED ROUGH DRAFT TRANSLATION

TRANSACTIONS OF THE INSTITUTE OF CONSTRUCTION AND CONSTRUCTION MATERIALS (SELECTED ARTICLES)

English Pages: 11

Source: Russian Book, Trudy Instituta Stroitel'stva i Stroymaterialov, No. 2, Izdatel'stvo Akademii Nauk SSR, Alma-Ata, 1959, pp. 163-166 and 179-182.

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Date 2 May 1963

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# THE PROBLEM OF THE PRESENCE OF DEVITRITE IN FOAM GLASS*

R. L. Shuster, T. P. Polyakova

The optical constants and the structure of new crystalline formations in foam glass are not elucidated in the known literature; T. N. Keshish'yan and B. Long [1,2,3] indicate only the influence of the crystalline phase on the physicomechanical properties of foam glass.

As we know, the Ivotsk and Gomel'sk glass factories produce foam glass at optimum kiln temperatures of 900-920° and 700-720° respectively. For manufacture of ordinary glass, for example plate glass, these temperatures are not detrimental with respect to crystallization [4,5].

The crystallization process in foam glass depends on both the physical structure of the starting batch and the change of the chemical composition of the glass due to its interaction with a gasifier. Moreover, the decomposition products of the latter, for example limestone in a finely-dispersed state, "in statu nascendi", act as centers

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* This study was carried out in 1955.

for the formation of crystals of devitrite (a sodium calcium silicate) which under appropriate heat-treatment conditions cause complete crystallization of the entire metal of foam glass.

For petrographic and x-ray diffraction studies, we took samples of foam glass obtained by heating mixtures of glass powder (90%) and a gasifier (10% limestone or dolomite) to 860° with a holding at this temperature for five minutes.

During the microscopic study of foam glass microsections, crystals, intergrown into the groundmass of glass, were distinctly evident in almost all cases. We did not observe very many crystals in the glass when there was only a brief heat treatment of these batches or when they contained gasifiers — carbon-forming substances (coke, coal, peat fines).

The investigations of the mineralogical composition of foam glass, made by introducing limestone or dolomite permitted us to establish that the glass contained devitrite in the form of divergent-structural aggregates of acicular crystals whose index of refraction was $\alpha = 1.575$, $\gamma = 1.560$ [6] (see illustration).

The amount of devitrite in foam glass increases with an increase of heating temperature, increase in percentage of gasifiers (limestone or dolomite) in the mixture, and also with an increase of the specific surface of the glass powder and gasifier.

The new crystalline formations in foam glass were studied by petrographic and at the same time by x-ray diffraction methods.

X-ray photographs of artificially synthesized devitrite were investigated because there are not [7,8] standard values of the interplanar spacings and relative intensity of devitrite lines and also to confirming its presence in the foam glass.

According to the ternary phase diagram $Na_2O$ - CaO - $SiO_2$, the compound $Na_2O \cdot 3CaO \cdot 6SiO_2$ is the initial phase of industrial glass having the ingredients:

$SiO_2=66\div74\%$; $CaO=5\div17\%$; $Na_2O=10\div29\%$.

The stability field of devitrite is small and is set off from the point of the structure proper.

G. V. Morey and N. O. Boun [9] experimentally established that the product of the heat treatment of glass containing $NaO_2$ (10.5%), CaO (28.45%), and SiO (61.05%) i.e., corresponding to the molecular formula $Na_2O \cdot 3\ CaO \cdot 6SiO_2$, was comprised of crystals having the same optical properties as well-developed devitrite crystals.

To synthesize a glass whose composition corresponded to the forumla $Na_2O \cdot 3\ CaO \cdot 6\ SiO_2$, we used the following raw materials: quartz sand from the Mugodzhar deposit Kazakhstan ($SiO_2$— 99.2%; $Al_2O_3$ — 0.21%; CaO — 0.3%; $Fe_2O_3$ - 0.04%); soda ash of the "chemically pure" type ($Na_2O$ — 48.2%); and limestone from the Kaskelen deposit, Kazakhstan (CaO — 56%). The materials were ground in an agate mortar and mixed in the ratio of: sand 61.1 g, soda 21.8 g, limestone 50.8 g. Melting of the metal was done in a chamotte crucible housed in an oil kiln at a temperature of 1450° with a two-hour holding. The glass obtained was transparent, completely melted and fine. To accomplish the crystallization process the glass powder was heated at 95° for 3, 6, 9 and 12 hours. Opaque cores were obtained. When examining these under a microscope, we saw distinct accumulations of acicular crystals whose refractive index corresponded to that of devitrite.

In the chemical structure of the crystallized metal we noted 6% $Al_2O_3$ which is due to the permeation of aluminum oxide into the metal from the crucible body.

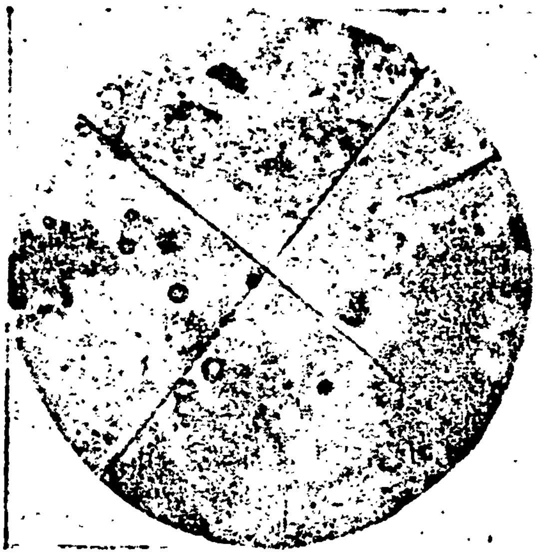

Fig. 1.

The obtained glass specimens were subjected to x-ray examination. The glass powders were placed in a small celluloid tube (0.7 mm in diameter) and were symmetrically set, according to the Debye method, in a powder camera: RPK-2 for No. 357 and RKD for No. 372 with a magazine radius of 28.65 mm. A special apparatus was the high-voltage source. An electron tube BCV-4 with a copper anode yielded the x-rays. The exposure regime was the same for all the studied samples: voltage 30 kv, current 15 milliamps, exposure 8 hours.

Filters were not used during exposure. The conditions of film developement werè uniform.

Visual examination of the x-ray photographs of the cake powders roasted with different holding times at 950° revealed the following: in spite of the background, the lines in the x-ray photographs were distinct and clear, generally at small angles. All the x-ray photographs appeared identical both in arrangement of lines and in their intensities. The most distinct and strongest lines were observed in the x-ray photograph of a core produced on heating a batch for 9 hours.

The second series of experiments was conducted with cakes produced by melting the indicated batches in special corundumized crucibles which eliminated the concentration of aluminum oxide in the metal. Quartz sand carefully freed of clay impurities was used. The conditions were the same during roentgenoscopic exposure of the crystallized metal. In addition, exposures were taken in iron and copper radiations with a nickel filter.

Visual examination of the x-ray photograph revealed that x-ray photographs also have a background, distinct and clear lines at small angles, and diffused lines at large angles.

We should note that the results obtained for one of these core, photographed with and without a filter, differ very little from each other.

The examination of the x-ray photographs for samples of the first and second founding showed that for the studied devitrite ($Na_2O$; $3CaO \cdot 6SiO_2$) Debye lines with a numerical value of the magnitude of the interplanary spacing equal to 3.03 A; 2.94 A – 1.7 9 A are characteristic. The comparison of the Debye powder patterns of synthetic devitrite and of foam glass of the indicated composition permitted us finally to verify the presence of devitrite in foam glass.

## Conclusion

1. The basic crystallization phase of foam glass obtained by applying limestone or dolomite as a gasifier, is devitrite.

2. The interplanary spacings of the most intense Debye rings of x-ray photographs of devitrite (see above) are characteristic for its identification.

3. The location of the indexes h, k, l and crystal lattice constants of devitrite (rhombic) requires further special investigation.

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# CONTENTS OF THE CRYSTALLINE PHASE IN FOAM GLASS IN RELATION TO CERTAIN FACTORS*

R. L. Shuster

From earlier studies [1, 2] we know that in almost all cases when examining foam glass under a microscope, the crystalline phase is clearly distinguishable along with the vitreous phase.

The problem of the composition and optical properties of new crystalline formations in foam glass is not elucidated in known literature. T. N. Keshish'yan [1,3] and B. Long [2] indicate only the influence of crystals in foam glass on certain of its properties: the compression strength of crystallized foam glass exceeds by 50% the same value for vitreous foam glass with equal bulk weight of both samples.

However, as T. N. Kechish'yan points out, with an increase of crystal content in foam glass, its moisture capacity significantly exceeds permissible limit.

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* This work was carried out with the advice of petrographer B. N. Karlysheva.

According to B. Long's data, to increase the degree of foam glass crystallization, it must be heated for a long time at the softening temperature.

The process of foam glass crystallization is intensified by two basic factors: by a content of numerous air bubbles in the walls of the formed pores which causes a significant accumulation of energy at the gas-glass interface and by the formation of a crystalline substance — decomposition products of the gasifier in a finely dispersed state which serves as a seed which under appropriate conditions can bring about the crystallization of the entire heated mass.

We investigated the problem of the influence of the composition of foam glass batches and their heat-treating conditions on the composition and on relative content of new crystalline formations in foam glass. For these objectives we used microscopic and x-ray diffraction methods [4].

Foam glass batches were prepared by mixing powder of window glass and a gasifier — limestone, dolomite or magnesite — in the amount of 0.5 to 10% of the glass weight.

## Methods of Investigation

The mineralogical composition of foam glass was studied under a microscope. Since new formations in foam glass are found as intergrown small crystals dispersed in the body of the glass, the method of extraction or partial exposure of these crystals was used to determine their optical constants. Taking into consideration the different solubilities of glass and crystals in a solution of caustic alkali, the powder of foam glass was heated for 30 hours in a water bath containing a solution of KOH; periodically, every 10 hours, the sediment was decanted and washed with hot distilled water. The powder, carefully prepared in this way, was investigated under a microscope; the results are presented below.

## The Investigation of Foam Glass with the Introduction of Limestone as a Gasifier.

The groundmass of foam glass obtained by heating the batch at 760° is a glass with solitary grains of nondissociated limestone, which owing to dissociation acquired a round shape which had begun and, obviously, to the interaction with the glass. The glass is permeated with radial-fibrous aggregates of acicular crystals 10 to 20 μ long, intergrown into the glass matrix; they sharply increase in number with an increase of heating temperature to 820°.

The optical properties of the crystals are characterized by direct extinction, positive lengthening and by an index of refraction equal to: $\alpha = 1.575$, $\gamma = 1.560$.

It has been established that the observed crystals are devitrite (Fig. 1).

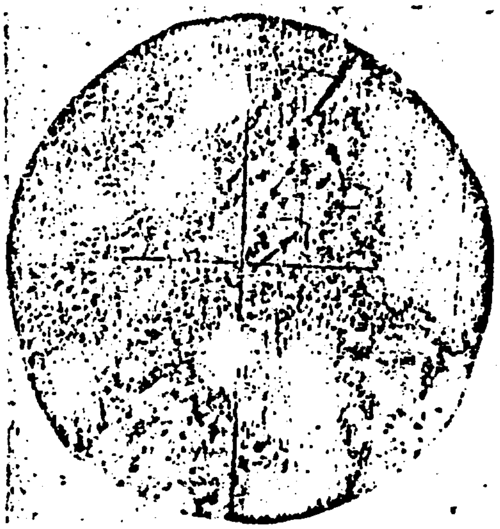

Fig. 1.

The study of foam glass samples from batches with a different specific surface both of the glass powder and of the gasifier indicates that the degree of crystallization here changes in direct proportion to the specific surface of the batch.

Thus, for example, in foam glass from batches of glass powder with a specific surface of 930 $cm^2/g$, very few devitrite crystals are found, whereas on heating a batch with a higher specific surface (3400 $cm^2/g$) under the same conditions the vitreous phase of foam glass appeared permeated with devitrite crystals grouped in radial-fibrous aggregates and clusters. The crystal lengths reached 20μ. The number of crystals increases with an increase of the specific surface of the gasifier. For example, with a decrease of the limestone particles from 200 to 30μ, the relative amount of the crystalline phase in foam glass increases approximately 5 times.

From the obtained data we can conclude: by increasing the specific surface of the batch components, mainly of the gasifier, we can increase the contents of the crystalline phase in foam glass.

This method is more economical in practice as compared with T. N. Keshish'yan's process which, for these purposes, increased the duration of heat treatment, thus increasing the moisture capacity of foam glass and impairing its quality.

## The Investigation of Foam Glass with the Introduction of Dolomite as a Gasifier

Samples of foam glass produced by heating batches to 820-860° containing a gasifier in the amount of 5 to 10% were subject to investigation. Under the microscope it was established that the foam glass contains crystals of devitrite, enstatite and diopside; the number and size of the enstatite and diopside crystals (as isometric microcrystals) increase somewhat with a change of heating temperature from 760 to 860° and of the content of dolomite in the mixture from 2 to 10% (Fig. 2).

The results of the determination of the phase composition of foam glass were confirmed by the data of x-ray diffraction examinations.*

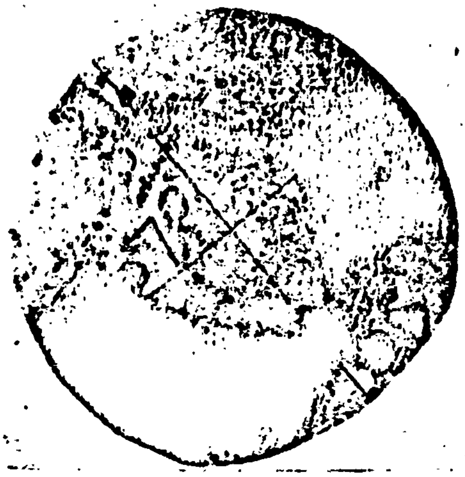

Fig. 2.

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* See in this collection, the article by R. L. Shuster and T. P. Polyakova "The Problem of the Presence of Devitrite in Foam Glass".

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