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Synthesis and study of garnet ceramic pigments

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Introduction. Ceramic pigments are inorganic colored finely dispersed powders which, when added to a material, impart certain color and change some of its properties. Garnets are a group of minerals different by composition but with analogous chemical formulae and similar appearance of their crystals. The transparent saturated colored garnets are demanded precious stones. The name of the group comes from the Latin word granatus which stands for the seeds of the granate tree. Garnets have various colors: purple red - almandine, colorless of yellow - green - grossular, brown or black - melanite, green - uvarovite, red - pyrope and andrdite, etc.

Materials and methods. For the preparation of garnet ceramic pigments in the system $CaO.Cr_2O_3.SiO_2$, the blends were defined on the basis of the stoichiometry of the main mineral – uvarovite $Ca_3Cr_2(SiO_4)_3$. The following composition was selected for the pigments - $3CaO.Cr_2O_3.3SiO_2$. The mineralizer used in the synthesis to decrease the synthesis temperature and accelerate the processes of formation of the new phase was H_3BO_3. The materials used for the synthesis were CaO, Cr_2O_3, $SiO_2.nH_2O$ and H_3BO_3. The substance used to introduce SiO_2 into the system - $SiO_2.nH_2O$, is much more reactive than the common quartz sand and the particle sizes were dispersed in the range 2-7μm. The quantities of the materials from which 100 g blend is prepared were weighed with precision of 0,1 g, then they were mixed and homogenized in dry state in a planetary mill Pulverizete - 6, product of "Fritch". The sintering was carried out in a laboratory muffle oven at heating rate 300 - 400°C/h in air atmosphere; the blend was placed in a porcelain crucible with a lid. The isothermal period at the final temperature was 2 hours. The pigments were sintered at 800°C, 900°C, 1000°C, 1100°C and 1200°C.

Results and discussion. X-ray phase analysis as a direct method for identification of phases. It is based on the diffraction of X-rays. The main task of the X-ray analysis was to identify the different phases individually or aim blends using the diffraction pattern registered from the sample studied.

The basic method of the phase analysis is the powder method which is widely used due to its simplicity and ease of versatility. The X-ray studies were performed on an apparatus IRIS with Cu K_α radiation and nickel filter, in the range of angles from 2 to 80°. The interplanar distances (d, nm) were calculated by the formula of Wulf - Bragg: $n.\lambda = 2d.\sin\theta$, where: λ – X-ray wavelength, nm; n – diffraction order (n = positive integer); θ – Bragg's angle of diffraction, grad. X-ray patterns of the garnet ceramic pigments synthesized are presented in Fig. 1.

The pigments synthesized had stable green color and significant formation of the main phase - the mineral uvarovite $Ca_3Cr_2(SiO_4)_3$, was observed at 1100°C, although reflexes from wollastonite ($CaSiO_3$), crystobalite (SiO_2) Cr_2O_3 were also observed.

The colors of the pigments were determined spectrometrically with a Tintometer RT 100 Colour. The highest amount of green color /- a*/ was found for the pigment synthesized at 1100°C.

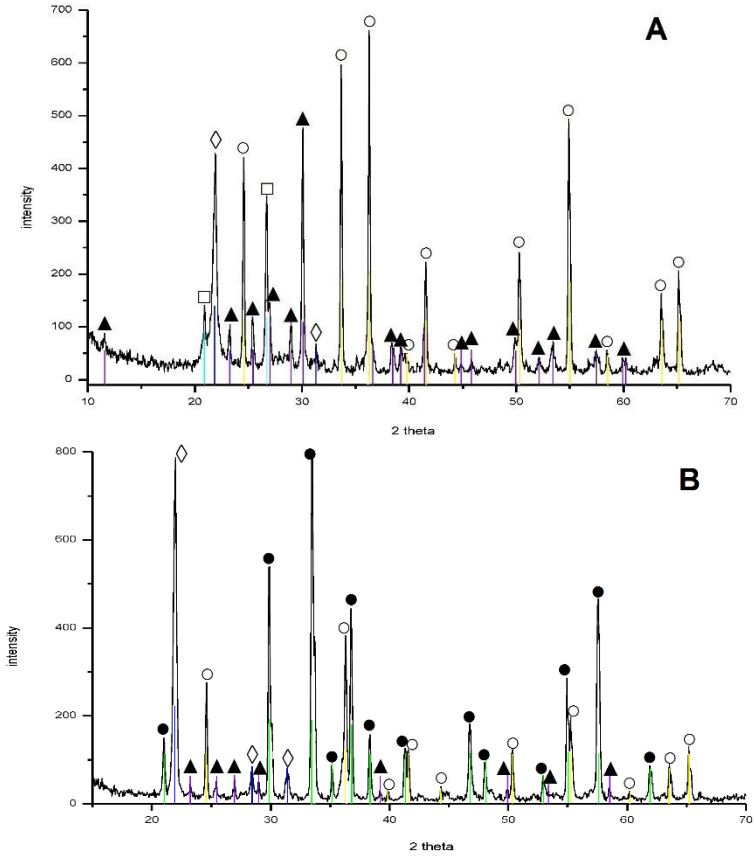

Fig.1 X-ray patterns of pigments in the system $3CaO.Cr_2O_3.3SiO_2$ taken at 900ºC (A), 1100 ºC (B)

● - Uvarovite $Ca_3Cr_2Si_3O_{12}$ ▲ - Wollastonite $CaSiO_3$ ◊ - Crystobalite SiO_2
□ - Quartz SiO_2 ○ - Chromium oxide Cr_2O_3

Conclusions. Green ceramic pigments were synthesized on the basis of the garnet uvarovite by the method of solid phase sintering. The optimal parameters of the process of synthesis were determined. The best results were obtained with the pigment synthesized at sintering temperature of 1100 ºC. The pigments obtained are suitable and can successfully be use in glazes for tiles and sanitary ceramics.

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