Kogarko L.N., Filina M.I., Bulatov V.K. Fractionation of pyroxen in the process of high-pressure differentiation of high-calcium larnit-normative melts close by composition to kimberlites. UDC 550.4:552.111

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**Abstract.** Obtained the first data on the composition of pyroxene in the process of crystallization of the larnitenormative turiaite, whose composition was similar to the natural dyke rock of the Maimecha-Kotui provincers up to 8 GPa. Experiments have shown that melilite nephelinite is characterized by very wide crystallization of melilite, clinopyroxene and olivine, which is a liquidus phase up to 30 kbar, which is significantly higher than the olivine stability field in tholeiitic and alkaline basalts of the same Mg number. At high pressures, the content of potassium and aluminum increases in pyroxenes, and the potassium distribution coefficient in pyroxenes increases.

Key words: kimberlite-like melts, phase equilibria, pyroxene, agpaitic magmas.

High-calcium unsaturated silica larnite-normative melts are the original magmas of ultrabasic-alkaline formations of the world, including melilitic rocks, ijolites, melteigites, carbonatites and nepheline syenites of the agpaitic and miaskite series. Phase equilibria during melting of larnite-normative melelite magmas have not been studied sufficiently, especially in the region of elevated pressures. In the literature, there are some data on the experimental modeling of melting and crystallization of melts with an increased concentration of calcium. However, they differ markedly from natural compounds.

In the largest in the world ultrabasic-alkaline province of Polar Siberia (Maimecha-Kotuiskaya), a powerful dyke series of undersaturated silicas of high-calcium magmas - melelite nephelinites, passing into kimberlites - is developed. Among these rocks, dykes of carbonatites and carbonate-containing phonolites and trachytes are very often encountered. In the present work, the composition of pyroxene in the process of crystallization of the larnite-normative turiaite, whose composition was similar to the natural dyke rock of the Maimecha-Kotui province, was studied for the first time in detail.

Experiments were carried out in aWalker-type multi-anvil apparatus with WC cubes with 8mm truncation edges. The pressure assembly consisted of a 95% MgO and 5%  $Cr_2O_3$  octahedron, a zirconia sleeve, a rhenium foil heater and MgO inserts. Temperature was measured with a W-Re thermocouple inserted through an  $Al_2O_3$  ceramic sleeve. The free space was filled with Ciatronics

cement. Pressure was calibrated at room temperature (Bi phasetransitions) and at high temperatures using the Mg<sub>2</sub>SiO<sub>4</sub> ransition at 1200  $^{0}$ C and 13-6 GPa (Morishima et al., 1994), the coesite-stishovitetransition at 1200  $^{0}$ C and 9-2 GPa (Yagi and Akimoto, 1976) and the CaGeO3 garnet-perovskite transition at 1100<sup>0</sup> Cand 6-1GPa (Susaki et al., 1985).The experiment was carried out at a pressure of 8 GPa and temperatures of 1300 °C and 800 °C.

For glass oxide studies, glasses of the given composition were synthesized. The necessary amounts of the starting material were placed in capsules made of graphite or spectrally pure iron, or together with distilled water and silver oxolate (a source of carbon dioxide) into platinum and silverpalladium ampoules in the case of experiments with volatile components. Data from experiments carried out under dry conditions made it possible to construct a phase equilibrium diagram of melilite nepheline in coordinate temperature-pressure. Experiments have shown that melilith nepheline is characterized by very wide crystallization of melilite, clinopyroxene and olivine, which is a liquidus phase up to 30 kbar, which is significantly higher than the olivine stability field in tholeiitic and alkaline basalts of the same magnesia.

To assess the evolution of pyroxenes was used the Na-Mg parameter proposed by Stephenson (1972) (Fig. 1), is based on the atomic proportions of the elements. In the process of crystallization, the composition of pyroxene changes; as the temperature increases, the contents of aluminum and sodium increase in pyroxenes, while the concentrations of silicon, calcium, magnesium and iron tend to decrease.

Deep high-temperature differentiation of magmatic high-calcium melts will lead to very rapid depletion of residual liquids by aluminum and the formation of supersaturated liquids. At high pressures, the content of potassium and aluminum increases in pyroxenes, and the potassium distribution coefficient in pyroxenes increases.

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Fig. 1 Variations in the composition of pyroxenes with respect to the (Na-Mg) fractionation index, shown as cationic proportions per 6 oxygen atoms.

# Dushenko N.V., Dnestrovskii A.Yu., Voropaev S.A. Experimental investigations of cavitation as a possible process of synthesis of diamond in nature.

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**Abstract.** The experimental setup using hydrodynamic cavitation (CNDS) at the continuous cycle was constructed at GEOKHI RAS. The features of the carbon nanoparticles formation by means of cavitation using hydraulic shock are considered., Crystal structures of various synthesized carbon nanoclusters during cavitation in 1% solution of isopropyl alcohol ( $C_3H_7OH$ ) in water are studied with method of electronic diffraction. Nanodiamond diffraction structures are obtained.

Keywords: Cavitation, hydroshock, diamond, carbon, nanoparticles

This work is a continuation of experiments on the cavitation synthesis of carbon nanostructures in GEOKHI RAS. For the first time, the hypothesis of obtaining nanodiamonds during cavitation in the currents of carbon-containing liquids were made by academician E. M. Galimov [Galimov, 1973]. At the

beginning of 2000-ies in the N. E. Bauman Moscow State Technical University MSTU jointly with the group of professor V. N. Skorobogatskiy the development of an experimental setup of the cavitation synthesis was carried out. In 2004 the first experimental confirmation of the hypothesis of academician Galimov E. M. [Galimov et al, 2004] was obtained. Further experiments on this device were carried out with various carbon-containing liquids: benzene, toluene, in a mixture of ethanolaniline [Voropaev et al., 2011, 2012, 2014], which showed the production of the diamond structure in these liquids during cavitation. However, this unit had several drawbacks: an experimental method includes the powder explosion and a single time operation cycle. In 2016, in GEOKHI RAS under the guidance of academician Galimov E. M. a new Cavitation synthesis of NanoDiamonds Facility (CNDF) with continuous cycle was created. This paper provides a description of this device and presents first results.

CNDF works on the principle of the hydraulic impact: pump pushes the carbon-containing fluid along the contour of a closed system, in the shaped

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nozzle the formation of cavitation bubbles occurs and with a certain frequency (about 3 times per minute) the flow of fluid is blocked, causing the hydraulic impact at bubbles. For this purpose the original design of a cyclically repeatable hydraulic impact was created with a quick-closing pneumatic valve that contains an electronic timer of opening and closing the valve. The pneumatic valve consists of a ball valve with mounted actuator, a directional valve, dumper mufflers and compressor stations. In the working circuit, consisting of two parallel pipes, the bypass pipe serves as a safety channel in order to avoid the disruption of the setting circuit. Manual valve in bypass pipe allows to control the flow velocity in the working channel and intends to reflect the shock wave arising from hydroimpact.



The schematic diagram of CDNF is shown in Fig. 1 [Dushenko et al., 2016]. The following parts are used: centrifugal pump ( $P_{max} = 5$  atm) functioning under the control of an electronic pressure sensor and electronic control unit (frequency converter), the working circuit consisting of two parallel tubes: bypass tube with the manual valve and working channel with a profiled cavitator nozzle and a quick-closing pneumatic valve under the control of compressor station ( $P = 8 \div 10$  atm) and electronic timer, tank mounted with the relief valve, system pressure gauges of MT-40 mechanical type.

Series of calculations with the hydrodynamic module ANSYS CFX-physical code for selecting the optimal profile of the nozzle and the operating parameters of the experimental setup CNDF were carried out [Dnestrovskii et al., 2017]. A number of calculations of a single-phase modeling as well as a hydraulic impact in two-phase fluid modelling were performed. As a result of calculations it was found that at a given operating pressure of the pump  $P_{in} =$ 4.6 atm at the time of hydraulic impact the pressure jump in the liquid reaches  $P_{in} = 9.0$  atm.

Measurements showed that the pressure jump during the hydraulic impact in CNDF reaches 10 ATM. As expected according to the calculations [Dnestrovskii et al., 2016] with such an external pressure the shock wave formed inside the bubble as a result of a number of fast chemical and physical processes produces values of temperature and pressure high enough to form the solid diamond phase with a size of 5 nm.



**Fig. 2.** Photo picture obtained by the electron microscope: nanodiamonds from isopropyl alcohol (enlargement by 100000, the particle size of 3-5nm)

Experiments on CNDF was carried out with 1% solution of IsoPropyl Alcohol ( $C_3H_7OH$ ) in water (IPA). After the chemical cleaning of the fluid [Voropaev et al., 2011] the resulting material was investigated by transmission electron microscope JEM100CX11 in the ISSP RAS. Fig. 2 shows a photograph of samples of cavitation nanodiamonds obtained from IPA.

Nanodiamond particles are gray clusters in nanometer range. There are also seen black particles in Fig.2 that represents a compressed amorphous carbon formation. The size of the diamonds from the isopropyl alcohol is on our estimate of 3-5 nm, which is slightly smaller than 10-30 nm nanodiamonds obtained in toluene by the same mechanism [Voropaev et al., 2012]. Fig. 3 shows the electron

diffraction pattern obtained by an electronic microscope and confirming the nature of the diamond nanoparticles from IPA. Table. 1 shows the results of processing of electronograms of IPA samples, and includes the experimental data obtained in a pilot plant of MSTU device with benzene [Voropaev et al., 2011], toluene [Voropayev et al., 2012] and the ethanol-aniline [Voropyev et al., 2014] and tabular data for crystalline diamond.

When comparing results of electronogram patterns with tabular values a good agreement is obtained in all cases. But there are lines (200 and 240 hkl hkl) for sample No. 52, Fig.3A, corresponding to the interplanar distance of 1.78 A and 0.7976 A. These lines are forbidden for the diamond lattice (space group Fd3m), but allowed for the FCC lattice (space group Fm3m). Forbidden lines correspond to reflections from planes (200) and (240) cubic lattice with the same constant, as in the diamond lattice a = 2,563 A. These lines exist in many electron-diffraction patterns of the cavitation diamonds. From

the analysis of electronograms patterns, we can conclude that the forbidden line is more intense in the case of larger particles (electron-diffraction patterns are point reflexes), which confirms the presence of a mixture of different crystalline phases – a conventional diamond phase and FCC phase of carbon. Table 1 also shows that the line of sample No. 49, Fig.3b, coincide with a tabular value of a diamond and do not contain any forbidden lines, which is a sign of a pure diamond structure of this sample.

It is shown that CNDF device with hydraulic impact mechanism, gives the pressure jump of the order of 10 atm. With this pressure it is possible to synthesize diamond phase in the carbon-containing liquids. On the other hand, the presence of distortions in a diamond structure allows to make a conclusion about the insufficient value of this pressure jump. We hope to increase the pressure up to 20 atm by optimizing the configuration of the valves.





Fig. 3. The electron diffraction patterns obtained by the electron transmission microscope for particles obtained in IPA experiments.

Table	1. The interplanar space	ing from the processing	g of electron diffr	raction patterns in Fi	g. 3. Normalization to
plane	111, "-" - lines not dete	cted			

		Standard Experimental installation of the MSTU. NE Bauman				Experimental installation GEOKHI RAS	
The line number		FCC lattice Diamond,	Cav. Diamond from benzene, A	Cav. Diamond, from toluene, A [Voropaev et	Cav. Diamond, from ethanol-	Cav. Diamond, from mixture of 1% IPS in water, A	
and plan (hkl index	plane ndex)	A	[Voropaev et al., 2011].	al., 2012].	aniline, A [Voropaev et al., 2014].	№52 Fig 3a	№49 Fig. 3b
1	111	2.06	2.06	2.06	2,06	2.06	2,06
2	$200^*$	(1.783)	1.79	1.85	1,74-1,78	1.782	-
3	220	1.261	1.26	1.26	1,25-1,28	1.21	1,256
4	311	1.0754	1.08	1.076	1,08-1,11	-	1,06
5	222	1.0297	-	-	-	1.04	-
6	331	0.8182	0.821	0.814	-	-	0,81
7	240*	(0.7976)	-	-	-	0.79	-
8	422	0.7281	-	-	-	0.744	0,724
9	115	0.6864	-	-	-	0.692	-

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# Limanov E.V.<sup>1,2</sup>, Varlamov D.A.<sup>1</sup>, Kuzyura A.V.<sup>1</sup>, Litvin Yu.A.<sup>1</sup>Garnetization of olivine in experiment – model of ultrabasic-basic tranzition in the earth mantle.

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**Abstract.** The paper presents experimental results on transition from primitive garnet lherzolite of the upper mantle to eclogite that was reproduced in physico-chemical experiment at diamond formation parameters. The evolution of ultrabasic paragenesis to basic one became possible due to two peritectic reactions: one of them concerns with a disappearance of orthopyroxene upon interaction with a silicate melt (Opx + L = Cpx), garnet appears as a result of the second reaction (olivine with jadeite). Thus, transitional associations up to pure eclogitic ones were obtained from the peridotite mixture OI – Cpx (=Di<sub>x</sub>Jd<sub>1-x</sub>) in various proportions of the starting components.

Keywords: fractional crystallization, eclogite, peridotite, mantle-carbonatite concept, mantle, partial melting

The rocks of the upper mantle are differentiated and are represented mainly by ultrabasic garnet peridotites and pyroxenites, which dominate over eclogites at 95:5 vol.%, respectively (Ringwood, 1975). Occasionally (about 2%) diamondiferous peridotites, pyroxenites, eclogites, and grospidites are found along with non-diamondiferous rocks among the upper mantle xenoliths (Sobolev, 1974; Dawson, 1983; Spetsius, Taylor, 2008). At the same time, diamondiferous eclogites among mantle xenoliths are 1-2 orders of magnitude greater than peridotite ones. Generalization of analytical data on chemical and phase compositions of rock-forming minerals of the upper mantle and paragenetic inclusions in diamonds (Litvin, 1991) shows that they belong to the single multicomponent olivine complex system [O] =  $(Mg,Fe)_2SiO_4$ ] - garnet [Grt =  $(Mg, Fe)_3Al_2Si_3O_{12}$ ] clinopyroxene [Cpx = [Ca (Mg, Fe)  $Si_2O6$  ·  $NaAlSi_2O_6]_{ss}$ ] - corundum [Crn =  $Al_2O_3$ ] - coesite  $[Coes = SiO_2]$ . The diagram-complex of the compositions of the garnet-peridotite facies of the upper mantle (Fig. 1, Litvin et al., 2016) consists of two ultrabasic ("olivine-saturated") simplex diagrams: (1) of peridotite-pyroxenites (Ol-Opx-Grt-Cpx) and (2) of olivine eclogites (Ol-Grt-Cpx-Crn), and also the 3-basic ("silica-saturated") simplex diagrams: (3) of corundum-kyanite eclogites (Grt-Cpx-Crn-Ky), (4) of kyanite-coesite eclogites (Grt-Cpx-Ky-Coes) and (5) of coesite-orthopyroxene eclogites (Grt-Cpx-Coes-Opx). This allows us to investigate the ultrabasic-basic petrological and mineralogical objects of the upper mantle within the framework of a single physicochemical system (Fig. 2).



**Fig. 1.** Phase relations of the peridotite-eclogite system Ol-Cpx-Crn-Coe of the upper mantle (Litvin et al., 2016)



**Fig. 2.** The complex liquidus of the peridotite-eclogite system Ol-Cpx-Crn-Coe in the projection on the divariant section of Ol, Cpx-Crn, Cpx-Coe, Cpx (Litvin et al., 2016)

The thermal barriers located on the planes of Ky-Grt-Cpx and Opx-Crn-Cpx fields are characterized by maximum temperature values for evolving melts. The second thermal barrier is known as the "eclogite thermal barrier" (O'Hara, 1968) indicating the impossibility of transition from ultrabasic to basic However, if the peridotite-pyroxenite rocks. composition of olivine-saturated rocks fall under changes and olivine and orthopyroxene is lost during peritectic reactions, then the figurative point of the composition of the Ol-Opx-Cpx-Grt field will shift to the Grt-Cpx line of bimineral eclogites. From there the point of the composition will be able to move further into the area of basic eclogites due to the saturation of residual melts with a jadeite component, with a transition of the system composition from olivine-saturated rocks to silica-saturated, which allows ultrabasic-basic evolution to have place. A physico-chemical possibility of the paragenetic peridotite-eclogite transition can be realized as a result of fractional evolution of a substance of primitive garnet lherzolite of the upper mantle.

Experimental studies of phase ratios at melting of the ultrabasic peridotite-pyroxenite system under conditions of the garnet-peridotite facies of the upper mantle (Litvin, 1991) found out that it is characterized by peritectic nonvariant subsolidus association L + Ol + Opx + Cpx + Grt (the clue peritectic reaction  $L + Opx \rightarrow Cpx$  with the disappearance of Opx), after which with decreasing temperature the stable monovariant equilibrium L +Ol + Cpx + Grt passing through the "piercing point" into the adjacent olivine-eclogite simplex is stable. It was experimentally established that at high pressure (above 4.5 GPa) olivine is processed into a garnet in a reaction with jadeite component (Gasparik, Litvin, 1997). In further studies, the reaction of olivine garnetization was purposefully studied in the model system Fo-Di-Jd (Butvina, Litvin, 2009).

In the present paper the variable contents of jadeite component in the Ol-Cpx (=  $Di_XJd_{1-X}$ ) system studied in equilibrium approximation are considered, that allow to estimate its physicochemical behavior in a regime of fractional crystallization.

The diagram of the liquidus surface Ol-Jd-Di (Fig. 3) is constructed (Fig. 3), where "Cpx 327" is a particular experimental composition of peridotite clinopyroxene close to natural one (Litvin, 1991). The experiments were carried out at a pressure of 6 GPa and a temperature range from 1200 to 1650°C. The liquidus surface diagram consists of the liquidus fields Ol + L, Grt + L and Cpx + L, the monovariant curves Ol + Cpx + L, Ol + Grt + L and Cpx + Grt +L, and the quasi-non-variant peritectic point Ol + Cpx + Grt + L (effect of "olivine garnetization"). The position of the non-variant peritectic point on the liquidus of the Ol-Di-Jd system corresponds to the composition (in wt%): SiO<sub>2</sub> 47.1, Al<sub>2</sub>O<sub>3</sub> 2.75, MgO 33.5, FeO 8.75, CaO 6.15, Na<sub>2</sub>O 1.75, which also characterizes the peritectic melt composition.

At temperature decreasing the figurative point of olivine-saturated melt moves along the liquidus surface in the following order: the divariant field Ol + L  $\rightarrow$  the monovariant cotectic Ol + Grt + L  $\rightarrow$  the quasi-non-variant peritectic Ol + Grt + Cpx / Omph + L (Ol disappears)  $\rightarrow$  monovariant cotectic Omph+Grt+L, finish of crystallization and formation of bimineral eclogites Omph + Grt.



**Fig. 3.** The projection of liquidus surface of the Ol-Cpx system (=  $Di_x Jd_{1-x}$ ). P - peritectic point

The polythermal sections Ol -  $Cpx_{50}Jd_{50}$  (Fig. 4),  $Cpx_{60}Jd_{40}$  -  $Ol_{60}Jd_{40}$  (Fig. 5) are constructed, they reveal the peritectic physicochemical mechanism of Ol dissapearing in the 4-phase peritectic point Ol + Cpx + Grt + L (Fig. 2), which also demonstrate a change in total system composition during fractional

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crystallization. Simultaneously with olivine garnetization enrichment of clinopyroxene with a jadeite component occurs, with the conversion of ultrabasic Cpx into a basic omphacite Omph. The transition from the peritectic point Ol + Cpx + Grt + L (Fig. 2) to the conjugate simplexes of corundum-kyanite or kyanite-coesite or coesite-orthopyroxene T, °C



Fig. 4. The polythermal section of  $Cpx_{50}Jd_{50}$ -Ol of the Ol-Cpx(=  $Di_XJd_{1-X}$ ) system. P is a peritectic reaction of disappearing of olivine, characterizing by a transition from Cpx + Ol + Grt association of olivine eclogites to Cpx + Grt association of bimineral eclogites in the subsolidus

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eclogites, the corresponding differences in eclogite rocks can be formed.

Thus, the carried out studies demonstrate a possibility of ultrabasic-basic evolution under the conditions of the upper mantle, which indicates the real ability of its realization in natural matter.



Fig. 5. The polythermal section of  $Cpx_{50}Jd_{50}$  - Ol of the Ol-Cpx(= Di<sub>x</sub>Jd<sub>1-x</sub>) system. P is a peritectic reaction of disappearing of olivine, characterizing by a transition from Cpx + Ol + Grt association of olivine eclogites to the Cpx + Grt association of bimineral eclogites in the subsolidus.