

# The effect of composition of mantle fluids/melts on diamond formation processes

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## ABSTRACT

Experimental modeling of natural diamond-forming processes is performed at high  $P$ – $T$  conditions in the  $K_2CO_3$ – $Mg_2SiO_4$ – $KCl$ – $H_2O$ – $C$  system. In order to establish the influence of the crystallization environment composition on diamond nucleation and growth, most of the experiments were performed at fixed  $P$ ,  $T$ ,  $\tau$  parameters ( $P = 7.5$  GPa,  $T = 1600$  °C,  $\tau = 15$  h) and only the compositions of melts and fluids was varied. The degree of the graphite-to-diamond conversion ( $\alpha$ ), which is the quantitative indicator of diamond-forming capability of the medium, was measured in each experiment. The results of the present study show that in fluid-free carbonate, carbonate–silicate and carbonate–chloride melts diamond crystallize mainly by a FG (Film Growth) mechanism. In  $H_2O$ -containing melts and fluids only the TGG (Temperature Gradient Growth) mechanism of diamond crystallization was observed. Increasing water content in all water-containing melts and fluids of the studied system considerably enhances diamond nucleation and crystallization in the following sequence  $K_2CO_3$ – $H_2O > KCl$ – $H_2O > Mg_2SiO_4$ – $H_2O$ . The intensity of formation of metastable graphite increases with decreasing temperature and  $H_2O$  content. It was shown, that diamond crystallization mechanism similar to TGG can have more considerable distribution in nature, than FG. Based on the experimental data we considered possible scenarios of diamond and graphite behavior in nature. In the closed systems with excess carbon a decrease in temperature leads to diamond crystallization changes with joint crystallization of diamond and graphite, and afterward only metastable graphite crystallization takes place. At carbon excess the increasing of  $P$  and  $T$  lead to increasing of the intensity of diamond crystallization right up to formation of polycrystalline aggregates, and at deficiency in carbon only carbon dissolution occurs.

In the open systems without the additional source of carbon reducing of  $H_2O$  content and changing media from carbon-saturated aqueous fluid to fluid-free melt result in first to the diamond nucleation, then in the joint crystallization of diamond and graphite and finally in FG diamond synthesis. The evolution of media composition from fluid-free melt to aqueous fluid at the constant excess of carbon results in FG diamond synthesis, then joint crystallization of diamond and graphite and, finally, diamond nucleation similar to TGG mechanism.

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## 1. Introduction

The composition of the natural diamond crystallization environment is one of the principal questions in the problem of diamond genesis. The main source of information about the composition of the crystallization medium are the mineral and fluid inclusions in diamond. The mineral inclusions allow determining the diamond paragenesis and make possible estimation of  $P$ – $T$ – $f_{O_2}$  conditions (Sobolev, 1977; Meyer, 1987; Harris, 1992; Sobolev et al., 2001; Taylor et al., 2003; Sobolev et al., 2004; Stachel and Harris, 2008; Foley, 2008; Van Rythoven and Schulze, this issue; Sobolev et al., this issue). The fluid inclusions provide direct information about the composition of the crystallization medium (Navon et al., 1988; Schrauder and Navon, 1994; Navon, 1999; Izraeli et al., 2001). Analysis of data on micro- and nano-inclusions in diamonds from different localities, including mantle-derived (Klein-BenDavid

et al., 2004; Zedgenizov et al., 2004; Tomlinson et al., 2005; Klein-BenDavid et al., 2006; Tomlinson et al., 2006; Klein-BenDavid et al., 2007; Logvinova et al., 2008) and UHP metamorphic diamonds (De Corte et al., 1998; Dobrzhinetskaya et al., 2003; Hwang et al., 2005, 2006; Dobrzhinetskaya et al., 2007), indicates that the diamond-forming media at the stage of inclusion entrapment constituted super-critical fluid or melt with different ratios of carbonates, silicates, chlorides and water (Zedgenizov et al., this issue; Weiss et al., this issue; Klein-BenDavid et al., this issue). Detailed studies of inclusions in different growth zones of some crystals demonstrated considerable variations of the composition of diamond-forming media during the growth of individual crystals (Tomilenko et al., 2001; Klein-BenDavid et al., 2004; Shiryayev et al., 2005; Weiss et al., 2008). Moreover, in some cases isotopic composition and nitrogen content in diamonds were found to correlate with the composition of inclusions.

Many debatable questions concerning diamond genesis and, mainly, revealing the systematics between the media composition and the processes of diamond nucleation, growth and dissolution

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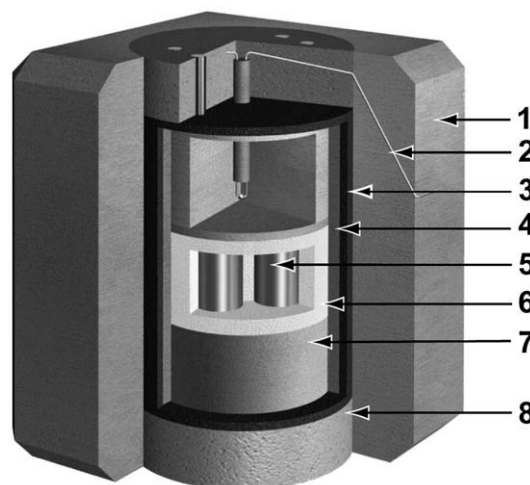
can be clarified applying experimental modeling. However, the experimental data available so far are insufficient to explain complicated systematics of diamond formation in nature. Diamond crystallization in the fluid-free alkaline carbonate melts has been studied in great details (Akaishi et al., 1990; Kanda et al., 1990; Taniguchi et al., 1996; Litvin et al., 1997; Pal'yanov et al., 1998, 2002). First data on diamond synthesis in carbonate–silicate melt (kimberlite) at 7–7.7 GPa and 1800–2200 °C were reported by Arima et al. (1993). Subsequent studies showed the possibility of reducing  $P$ – $T$  parameters of diamond crystallization in the carbonate–silicate melts (Shatskii et al., 2002; Pal'yanov et al., 2005a,b, 2007a) and extended substantially the range of compositions (Litvin and Zharikov, 2000; Litvin et al., 2008). Methodical features of many works consisted in mixing of initial powder reagents of carbonates, silicates and graphite. Such an approach allows realization of spontaneous crystallization of diamond in short experiments, but gives little information about possible mechanisms and systematics of diamond formation. The systematics and mechanisms of diamond crystallization in water-containing carbonate, silicate and chloride melts and fluids are of particular interest because modern concepts of natural diamond-forming media are based on the role of fluids (Haggerty, 1986; Deines and Harris, 1995; Navon, 1999; Taylor and Anand, 2004). For convenience of description, the media formed in the system studied will be divided into two groups: hydrous melts and aqueous fluids. Under conditions higher than the second critical end point (Wyllie and Ryabchikov, 2000) we will refer to water-poor and water-rich compositions as  $H_2O$  containing melts and aqueous fluids respectively. The processes of diamond crystallization in fluid and fluid containing environments have been studied in less detail. It is caused by the necessity of applying more complex method for realization of such experiments, particularly, the use of hermetic capsules. First experiments on diamond crystallization in the  $H_2O$ –C system were realized at 7.7 GPa and 2000–2200 °C (Yamaoka et al., 1992). Further investigations of diamond formation in the C–O–H system permitted to substantiate the existence of the induction period, which precedes diamond nucleation, and whose duration significantly increases with decreasing temperature (Akaishi and Yamaoka, 2000; Pal'yanov et al., 2000; Sokol et al., 2001; Yamaoka et al., 2002). It was experimentally shown, that moderately oxidized fluids of the C–O–H system are more favorable for diamond crystallization than reduced fluids (Sokol et al., 2004; Sokol and Pal'yanov, 2004). It was established, that addition of  $H_2O$  renders silicate media diamond-forming (Borz dov et al., 1999) and allows to decrease significantly the  $P$ – $T$  parameters of diamond crystallization in carbonate (Sokol et al., 2001; Pal'yanov et al., 2002) and chloride (Palyanov et al., 2007b) media. Only recently studies on diamond crystallization depending on the solid/fluid ratio have started (Palyanov et al., 2007b; Sokol and Pal'yanov, 2008).

The determining role of fluids in the process of diamond genesis and the wide variations of the composition of the mantle diamond-forming media, reconstructed based on studies of inclusions, require more detailed experimental investigations on diamond synthesis.

The aim of the present study is to perform a comparative analysis of experimental data on diamond crystallization in the model system  $K_2CO_3$ – $Mg_2SiO_4$ – $KCl$ – $H_2O$ –C, to establish the main mechanisms and systematics of diamond crystallization with reference to the medium composition and to interpret these data in accordance with possible scenarios of change of  $P$ – $T$  conditions and evolution of the composition of the mantle or UHP metamorphic fluids and melts.

## 2. Experimental procedure

Experiments were carried out using a multi-anvil apparatus of the “split-sphere” type with 300 mm outer diameter of the sphere. Multi-anvil sphere of 8/6 type consists of 8 steel truncated anvils, forming an octahedral cavity. An inner set of six tungsten carbide anvils is located in the octahedral cavity. The upper and bottom anvils have square



**Fig. 1.** High-pressure cell for experimental modeling of diamond crystallization. 1 –  $ZrO_2$  container, 2 – thermocouple, 3 – graphite heater, 4 –  $MgO$  sleeve, 5 – Pt capsules, 6 –  $CsCl$ , 7 –  $ZrO_2$ , 8 – talk ceramic.

truncation and four side anvils have rectangular truncation. The inner cavity formed by these anvils has a form of tetragonal prism. The corresponding high-pressure cell has sizes  $19 \times 19 \times 22$  mm and geometry as shown in Fig. 1. This high-pressure cell was calibrated for temperature and pressure with an account for heating. The results are given in details in the work of Pal'yanov et al. (2002). For experiments at 7 GPa and 1700 °C, the accuracy was estimated to be  $\pm 0.2$  GPa and  $\pm 40$  °C. Temperature was measured in each experiment with a PtRh30/PtRh6 thermocouple whose junction was placed inside the heater near the Pt capsules with the sample. To determine the effect of the medium composition on diamond crystallization more precisely, in the most of the experiments we used two capsules with different composition located symmetrically within the high-pressure cell, so that they were subjected to the exact same  $P$ ,  $T$  and  $DT$ . The elongated geometry of the cell makes it possible to use graphite heater 15.8 mm long and 9 mm in diameter. Two Pt capsules, each 3 mm in diameter and 4 mm long, were symmetrically pressed in  $CsCl$ . The ratio of the heater height (15.8 mm), and the height of capsules (4 mm), placed in the central low-gradient zone, provides temperature gradient not more than 10 °C/mm (Pal'yanov et al., 2002).

For experimental comparison of the efficiency of different media with respect to diamond crystallization, it is necessary to consider the criteria, that can be used for the comparison. These criteria can be the degree of the graphite-to-diamond conversion and minimal  $P$ – $T$  parameters of diamond nucleation. The degree of the graphite-to-diamond conversion ( $\alpha$ ) is defined as  $\alpha = M_{Dm} / [M_{Dm} + M_{Gr}] \cdot 100$ , where  $M_{Dm}$  is the mass of obtained diamond and  $M_{Gr}$  is the mass of residual graphite (Palyanov et al., 2007b). In some cases, when the conversion degree is low the comparison can be made by the density of nucleation centers. Performing experiments with different media it is important to develop and apply an appropriate crystallization scheme, as well as  $P$ ,  $T$  and duration ( $\tau$ ) of experiments, which provide the maximal information value of the results. Furthermore, the experimental technique and selected  $P$ ,  $T$  and  $\tau$  parameters have to be constant over the entire series of experiments to make the comparison of the results possible.

Concerning the crystallization schemes, to our opinion, the least informative is the scheme where the initial powders of graphite and solvent are uniformly mixed in the volume. In this scheme, thanks to the maximal contact area between graphite and melt, it is possible to achieve maximal conversion of graphite to diamond in short runs, but in this case it is difficult to compare different media, since neither the density of nucleation centers nor the degree of the graphite-to-diamond conversion can be determined. High  $P$ – $T$  parameters

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