

Studies of graphite transformation at cold compression

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Abstract. A possibility of formation of novel carbon phases from graphite at continuous exposure under pressures of 18 GPa to 45 GPa at room temperature was examined. Features in the pressure dependence of resistance as well as its relaxation times were found in the range 27–35 GPa. The scanning electron microscope image of the sample subjected to the pressure of 45 GPa shows the inclusion of a new phase, which did not disappear after removal of the load. However, the new phase is poorly seen in the pressure dependence of resistivity because of shunting by a large amount of non-transformed graphite.

1. Introduction

As it is well known, at normal pressure, graphite is the stable solid phase of carbon with the minimum Gibbs energy among other modifications. At higher pressures, diamond appears to be the next stable solid carbon phase with the minimum Gibbs energy (for example, see [1]). In this connection, many experiments were performed studying the graphite-to-diamond transformation at shock loading [2, 3].

Recent years, there have been numerous discussions about the structure of different possible modifications of cold compressed graphite, such as bct-C4, K4-, M-, H-, R-, S-, T-, W- and Z-carbon. A new phase of graphite at cold compression has been found in many experiments, such as optical microscopy, x-ray diffraction, Raman spectroscopy [4]. Nevertheless, the explanations of the nature of this phase are so far controversial [5]. Some experiments confirm that this phase has a monoclinic structure ascribed to the M-carbon.

2. Experimental

High pressures have been generated in the high pressure cell with synthetic carbonado-type diamond anvils [6]. The anvils are good conductors and can be used as electric contacts for resistivity measurements. The method used allows us to study the same sample at successive increasing and decreasing pressure and also to keep it loaded during a long time.

The estimation of pressure was proved previously at extensive studies of different materials in a wide range of temperatures and pressures. The samples were of ~ 0.2 mm diameter and of thickness from 10 to 30 μm . The measurement procedure was published in details in [7].

The resistance measurements were carried out step by step in cycles of loading-unloading at pressure range from 18 GPa to 45 GPa. The exposure time at each fixed value of pressure was twenty four hours. After pressure treatment, the samples were examined by means of the



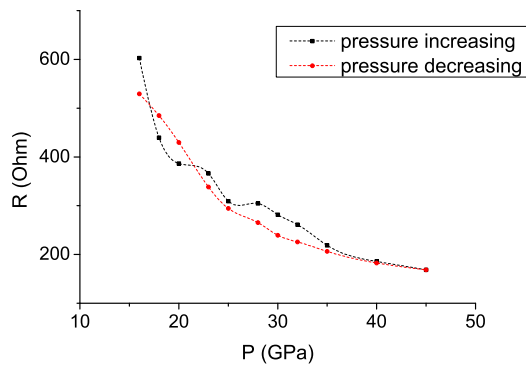


Figure 1. Pressure dependences of graphite resistivity at room temperature.

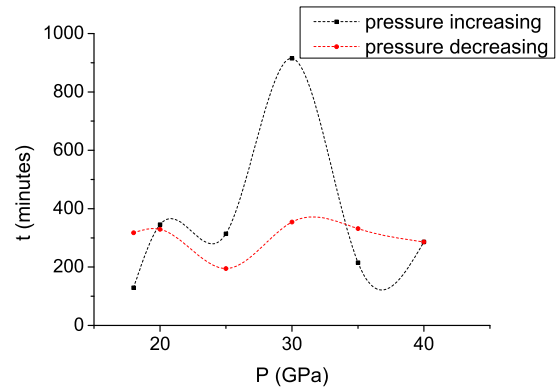


Figure 2. Pressure dependences of relaxation times of graphite resistivity.

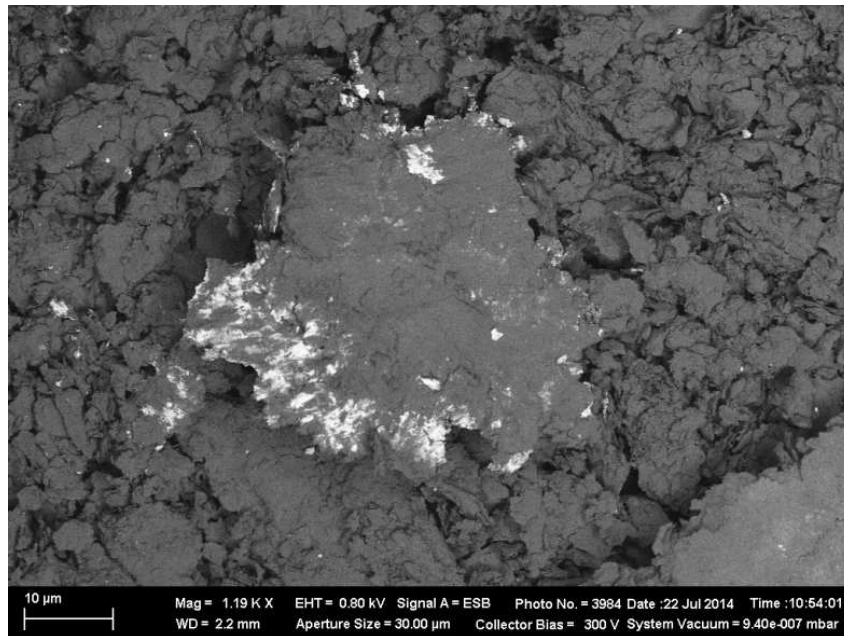


Figure 3. SEM image of the graphite sample after 24 hours exposure under 45 GPa.

workstation AURIGA CrossBeam, which is a scanning electron microscope with the possibility of x-ray microanalysis.

3. Results and discussion

The pressure dependence $R(P)$ of graphite is shown in figure 1. The samples were kept under stress at each pressure value during 30 min. Some features are seen at increasing pressure between 20 and 30 GPa. In principle, they can be associated with the formation of a new phase, however the features are weakly pronounced for such a conclusion, probably, because of shunting of sample resistance by a large remainder of non-transformed graphite.

The observed features become much more pronounced at increasing the time of exposure the samples under stress. The pressure dependence shown in figure 2 was obtained for the exposure time of twenty four hours at each fixed value of pressure.

The formation of new phase is confirmed by the workstation AURIGA CrossBeam, which is a scanning electron microscope (SEM) with the possibility of x-ray microanalysis. The SEM image of the sample subjected to the pressure of 45 GPa shows the inclusion of a new phase, which did not disappear after removal of the load (figure 3). It is shown by means of the microanalysis of the studied part of the sample that there are negligible inclusions of other chemical elements in the evenly distributed carbon phases.

4. Conclusion

The features in the pressure dependence of resistance as well as in its relaxation times were found in the range 27–35 GPa. These features were referred to new phase nucleation. The SEM image of the sample subjected to the pressure of 45 GPa shows the inclusion of a new phase, which did not disappear after removal of the load. However, the new phase is poorly seen in the pressure dependence of resistivity because of shunting by a large amount of non-transformed graphite.

Acknowledgments

This work was supported by the Government of the Sverdlovsk district and the RFBR (grant No. 13-02-96039).

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