Elecrochemical intercalation of fullerite C<sub>60</sub> using solid electrolytes Yu.A. Dobrovolskii,<sup>a</sup> L.S. Leonova,<sup>a</sup> E.V. Skokan,<sup>b</sup> I.V. Arkhangelskii,<sup>b</sup> and A.Ya. Borschevskii<sup>b</sup> <sup>a</sup> Institute of Problems of Chemical Physics, RAS, Chernogolovka, Moscow region, 142432, Russia <sup>b</sup> Department of Chemistry, Moscow State University, Moscow, 119899, Russia

The  $C_{60}$  crystals are known to have the closepacked face-centered lattice with relatively weak bonds between the molecules. In such a lattice, two tetrahedral and one octahedral sites per a molecule are available, therefore, fullerene appears to be suitable subject for intercalation.

Usually  $Me_xC_{60}$  compounds were synthesized by intercalation of  $C_{60}$  with alkali metals (Me) (or with their thermally unstable derivatives) in gas phase or in solutions [1, 2]; electrochemical intercalation from nonaqueous solutions was also used [3]. However, these methods did not allow one to control the extent of intercalation and to obtain individual compounds [4].

The aim of this work was to study electrochemical injection of the sodium ions from a solid electrolyte into fullerene: the processes on the electrode/electrolyte boundaries and the composition and properties of  $Me_xC_{60}$  compounds with x varying within a wide range were investigated.

The intercalation was carried out in a threeelectrode cell. Sodium-tungsten oxide bronze with the Na<sub>0.5</sub>WO<sub>3</sub> composition was used as a reference (RE) and working (WE) electrode and Nasicon was used as a solid electrolyte (SE). An electrochemical cell of the RE/SE/WE/SE/C<sub>60</sub> type was used. Injection was performed in galvanostatic conditions providing a voltage difference between WE and C<sub>60</sub>. A cell comprised of alternating cylindrical pellets of the electrolyte, electrodes, and fullerene pressed together. The amount of sodium intercalated was determined from the current flowed in the cell and the change in the voltage difference between WE and RE. The measurements were carried out under vacuum at 100-250 <sup>0</sup>C.

Since starting fullerene was dielectric, initially, the voltage of about 20 V was maintained for several hours until the current flowed in the cell exceeded 40 mkA ( $Na_xC_{60}$ , x<0.01). In this case, the electrolyte/fullerene boundary becomes reversible, and the e.m.f. between the RE and fullerene goes stable. Further, the voltage was decreased to maintain the constant current in the cell (see Fig.1)

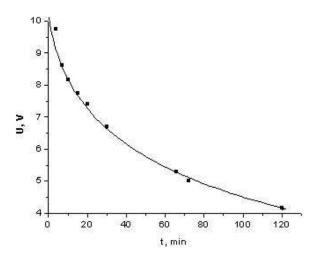


Fig.1. Voltage vs intercalation time (40 mkA)

After each changing in the composition of the samples by 0.05 mol, intercalation was stopped, and the open-chain cell was allowed to stand until the equilibrium was attained. Then, the impedance spectrum of the sample was determined.

Based on the data obtained, the intercalation mechanism and the structures of the compounds synthesized were discussed.

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 M.J.Rosseinsky // J.Mater.Chem., 5 (1995) 1497
H.H.Wang, J.A.Schuetter, A.A.Cooper and ... // J.Phys.Chem.Solids 54 (1993) 1655