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X-Ray Photoelectron Study of the Nanostructures Obtained from a Mixture of Metallurgical Powder (Ni, Fe) and Raw Polymer Materials

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Abstract—An X-ray photoelectron study of the mixtures of metal-containing materials from wastes of metallurgical production and raw polymer materials has been carried out. It is shown that the interaction of raw polymer materials with metallurgical powder may lead to metal reduction. The presence of reduced metal causes the formation of metal-containing carbon nanostructures.

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INTRODUCTION

Reprocessing of not only slags but also all other wastes (including powders), which makes the use of raw materials much more comprehensive, plays an important role in metallurgy. One of the methods for obtaining nanostructures is the synthesis of metal-containing carbon nanostructures during carbonization of raw polymer material.

In this paper, we report the results of X-ray photoelectron study of the possibility of applying metallurgical powder in synthesis of nanostructures.

EXPERIMENTAL

The study by X-ray photoelectron spectroscopy (XPS) were performed on an X-ray electron magnetic spectrometer with double focusing (resolution 0.1 eV, excitation in the AlK_α line (1486.6 eV), residual pressure 10^{-6} Pa) [1]

The XPS method was used to monitor the preparation of nanostructures from the relative content of C–C and C–H bonds, hybridization type of valence *s* and *p* electrons of carbon atoms and the satellite structure of the C1*s* spectrum, and the type of interaction of metal atoms with carbon. This method makes it possible to identify the C1*s* spectra and determine the chemical bonding of the elements, the nearest atomic environment, and the type of *sp* hybridization of valence electrons in nanostructures using the satellite structure of the C1*s* spectra. Application of 3*d* transition metal salts as catalysts in synthesis of nanostructures leads to the formation of C–C bonds with sp^2 and sp^3 hybridizations, whose ratio depends on the degree of occupying the *d* shell of the metal [2].

RESULTS AND DISCUSSION

A possible way of utilizing the metallurgical powder is its treatment by a polymer medium (polyvinyl alcohol (PVA)) and secondary polyvinylchloride (PVC) [3].

The study was performed on the following three samples. Sample no. 1 was the initial fine-grained metallurgical Ni-containing powder; sample no. 2 was prepared by mixing two solid components, fine-grained metallurgical Ni-containing powder and PVA, and heating the mixture to 400°C; and sample no. 3 was prepared by mixing a PVC solution in HCl and metallurgical Fe-containing powder, with subsequent heating to 400°C.

X-ray photoelectron spectra were measured for all samples. The full-range spectra of sample no. 1 of the initial fine-grained metallurgical Ni-containing powder exhibited C1*s*, O1*s*, and Ni3*p* lines.

The C1*s* spectrum (Fig. 1) has two components: C–H (285.0 eV) and C–O (287.0 eV), which indicate the presence of hydrocarbon contaminants in the metallurgical powder. The O1*s* spectrum also has two components, characteristic of metal oxides *Me*–O (529.5 eV) and adsorbed oxygen (532.0 eV). The Ni3*p* spectrum exhibits oxidized states of metals, i.e., the metal enters the composition of gas-dust ejections mainly in the form of oxides.

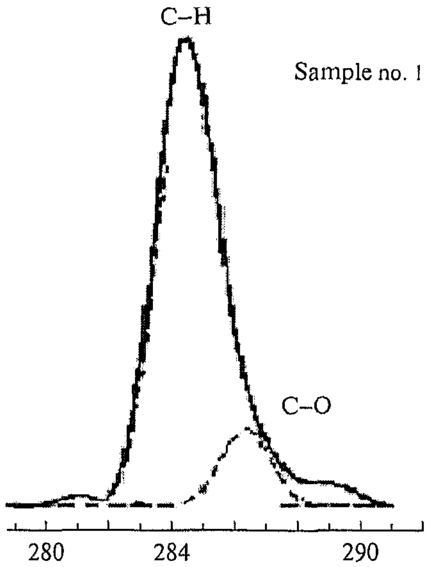

Fig. 1. X-ray photoelectron C1*s* spectra of samples no. 1.

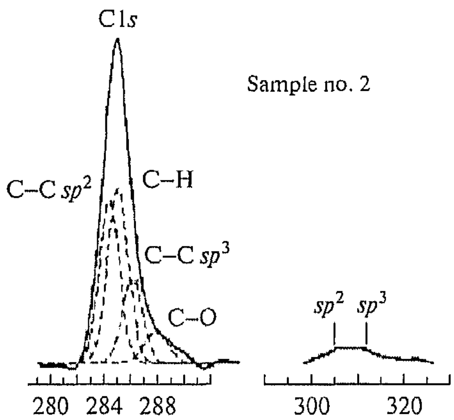

Fig. 2. X-ray photoelectron C1*s* spectra of samples no. 2.

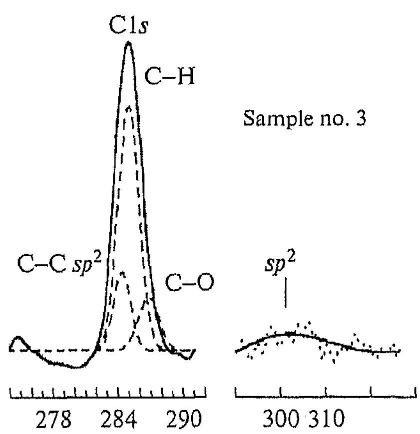

Fig. 3. X-ray photoelectron C1*s* spectra of samples no. 3.

The spectrum C1*s* of sample no. 2 (Fig. 2) demonstrates the presence of carbon not only in the C–H bond (285 eV) but also in the C–C bond with *sp* hybridization (284.3 eV) and C–C bond with sp^3 hybridization (286.1 eV). The changes in the Ni2*p* spectrum are indicative of partial reduction of nickel.

According to the data in the literature [4] on ultradispersed Ni powders with different particle sizes (from several tens to several hundreds Å), the thermal stability of NiO on particles of different size is different. On particles with sizes of several tens Å, NiO decomposes even at 200–250°C. On large (300 Å) particles, NiO is retained at temperatures up to T = 400°C. Such a difference is related to the smaller thickness of the oxide film (15 Å) on small particles; the film thickness on particles of several thousands Å in size increases by a factor of 1.5–2. Therefore, reduction of Ni from NiO requires lower temperatures in the case of nanoparticles.

Since iron is not reduced in PVA, we used secondary polyvinylchloride (PVC) dissolved in HCl as a raw polymer material. This allowed us to obtain graphite films, as evidenced by the presence of the component with sp^2 hybridization of electrons in the C1*s* spectrum (Fig. 3). In addition, the C1*s* spectrum contains C–H and C–O components. The Fe2*p* spectrum has a low intensity, which indicates that only iron traces are present in the samples under study. Iron is in the oxidized state. In contrast to the Ni-containing powder, study of the Fe-containing powder did not reveal metal reduction.

Thus, the X-ray photoelectron study of the samples obtained by heating mixtures of metal powder and raw polymer materials showed that metal reduction is observed only in the nickel-containing powder. Reduction of Ni from NiO requires lower temperatures in the case of nanoparticles. In contrast to the Ni-containing powder, metal is not reduced from the iron-containing powder.

CONCLUSIONS

The X-ray photoelectron study of the possibility of using metallurgical powder to form carbon nanostructures (with a metal compound as a necessary component involved in their formation) showed that an important factor is the metal ability to be reduced at certain temperatures. The interaction of PVA with metallurgical Ni-containing powder leads to metal reduction with the formation of both sp^2- and sp^3-hybridized structures, a fact indicating that carbon nanostructures are produced.

As a result of interaction of PVC with metallurgical Fe-containing powder, iron fails to be reduced to pure metal; however, the C1*s* spectrum contains a component with sp^2 hybridization of electrons, which is indicative of the formation of graphite-like structures.

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