

Interaction dynamics between compacted pyrophoric nickel nanopowders and air

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Experimental details

Nickel nanopowders were prepared as follows. Nickel formate was previously synthesized in the reaction of nickel hydroxide with formic acid. Synthesis of nickel hydroxide was carried out by alkaline treatment of nickel sulfate, precipitation and drying of the obtained hydroxide. The Ni formate begins to disproportionate at ~ 210 °C, forming a nickel nanopowder and releasing a gas mixture consisting of carbon dioxide, hydrogen and water vapour.^{S1} The reactor described previously^{S2,S3} with the sample of Ni formate powder 4 mm thick (15g) placed in a quartz trough, was kept in an electric furnace for 50 minutes at 209 °C in an argon stream. The samples were then treated with a hydrogen stream for 20 minutes, i.e. the total time spent by the sample in the furnace was 70 minutes. As a result, 4.7-4.8 g of pyrophoric nickel nanopowder was obtained; the powder samples were placed in small flasks in the medium of argon. The average diameter of the nanoparticles calculated from the specific surface area of the powder was 85 nm. The specific surface area of Ni nanoparticles was determined by BET method.

The opening of the flasks with nanopowder and all subsequent operations of weighing, pressing and measurement of sample sizes were carried out in a sealed box filled with inert gas (argon) and equipped with scales, press, measuring tools, presses and a gateway for changing powders and samples (Figure S1). The oxygen concentration in the box was monitored by the AKPM-1-02 analyser. When the flasks with powder were opened and the samples were pressed, the oxygen concentration in the box did not exceed 0.1 vol%. At the oxygen concentration, the nanopowders remained pyrophoric. Cylindrical compact samples with a diameter of 5 mm, 5.5÷13 mm in length and density of 1.5 ÷ 3.6 g/cm³ were obtained from the nanopowder. After pressing, each sample was placed in (a ground-glass stopper flask), and then removed from the sealed box by means of a lock.

The experimental investigation of heating, ignition and burning processes of the samples was carried out in the air. The samples were removed from the weighing bottle and mounted vertically for 3-5 seconds on a boron nitride stand. The change of temperature distribution within a sample surface in time and the determination of the maximum temperature were carried out with a Flir 60 infrared camera (60 frames/s, 320 x 240 pix, a sensitivity interval 8 ÷ 14 μm). A SONY FDR AX-700 video camera (50÷250 frames/s) was used to determine the combustion velocity (propagation of the oxidation reaction over the surface of the samples). The dynamics of temperature change within the sample surface was also controlled by two rolled thermocouples 40

μm thick, tightly touching the sample side surface. The thermocouples were located a millimeter and 4 mm from the bottom end of the sample.

The phase composition of the samples was studied by X-ray diffraction (XRD) by means of a diffractometer DRON-3M equipped with a source of monochromatic $\text{CuK}\alpha$ radiation. The recording of diffractograms was carried out in a step scanning mode in the range of angles $2\theta = 20 \div 80^\circ$ with a step 0.2° . The resulting diffractograms were analyzed by means of a PDF-2 database.

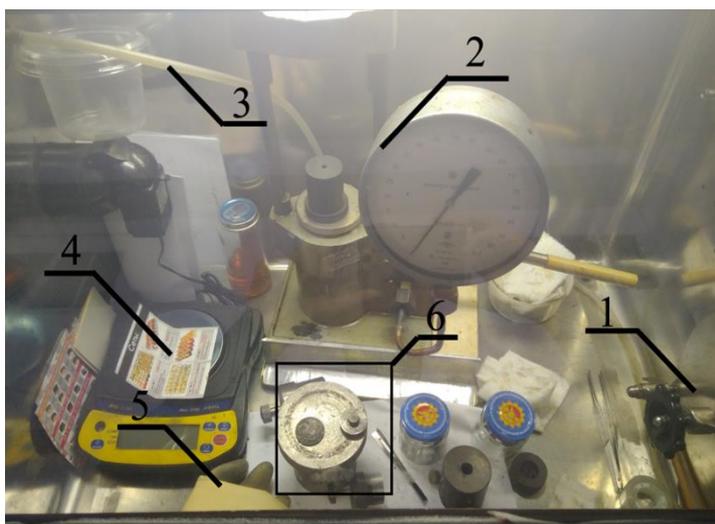


Figure S1 General view of the installation for obtaining compact samples inside the sealed box: (1) lock, (2) press, (3) output to the oxygen analyzer, (4) digital scales, (5) gloves and (6) set of presses.

References

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