Final reduction annealing of iron powders may prove to be particularly beneficial to them in the following three fields of application:

1) annealing as a preparatory operation designed to improve the physical and processing characteristics of powders before pressing;

2) reduction-decarburizing annealing of atomized iron powders as an essential part of the existing method of manufacture of powders by disintegration of liquid metals;

3) final, i.e., second (finishing), operation in a new, promising two-stage method of iron powder manufacture using direct iron production units [1].

In each of these three cases annealing constitutes the final operation imparting to the powder its ultimate properties, and its purpose is to ensure that a semifinished product is converted into a high-quality commercial iron powder. However, the existing solutions of this problem fail to meet the requirements of rational process control, and there is thus a need for further theoretical and experimental investigations leading to the development of an improved variant of the process and a more efficient plant [2].

Analysis of the existing industrial processes for the production of iron powder by the reduction of oxides [3] leads to the conclusion that the problem under consideration can be solved by subjecting a fluidized free-flowing charge to final reduction in a special unit under normal reducing gas pressure at moderate and elevated temperatures. Such an approach can markedly intensify heat and mass exchange processes and sharply decrease the time of the reduction operation, resulting in a fall in the cost of iron powder. Adopting this approach, the authors have developed a method for the heat treatment of powdered materials in vibrating bed furnaces [4, 5].

When low-frequency vibrations are applied to a reactor with a powder, the disperse material acquires a high-porosity structure, which is very useful in many processing operations. Vibratory fluidization prevents inhibition due to external diffusion and particle shielding and eliminates an undesirable phenomenon occurring in many powder heat treatment processes, viz., particle sintering and sticking to the reactor walls (crust formation). The process of gaseous metallic oxide reduction in a vibrating bed is an interesting subject for investigations in the field of topochemical reactions [6-8].

In the present work a flow-type apparatus was developed for the investigation of the final reduction annealing of iron powders (Fig. 1). The reactor 1 (in the apparatus there are two such reactors) is an oxidation-resistant steel tube placed in a heat-insulated shell 2. The shell is mounted on a vibrating platform 3 supported, via springs 4, on a massive base 15. A mechanical vibrator (a shaft with unbalanced weights) 5 has its own drive 6 and applies to the whole system vibrations with amplitudes of 0.5-4 mm and frequencies of 25, 33, and 42 Hz (curves 1-3 in Fig. 2) without change of drive belt transmission pulleys.

The reactor is heated from a welding transformer 7, which is connected to it through electric contacts 8 passing through the thermal insulation along the length of the reactor. With this arrangement of connections, it is quite easy to vary the length of the hot zone (up to 1600 mm). The provision of two reactors (each of which is heated by one of the secondary windings of the TS-500 welding transformer) enables parallel experiments to be conducted under identical conditions for two different states of the starting material and reducing gas and different rates of their supply to the reaction zone.
The material to be reduced is charged into a feeder 13, from which it descends into and advances along the preheated reactor under the action of vibration, passing through the required heat treatment cycle, the finished product falling into a vertical cooling collector 14. By suitable choice of vibration conditions and reactor slope, it is possible to regulate the speed of advance of the vibrating bed (Fig. 2), which in turn determines the time of residence of the powder in the hot zone (1-15 min) and the rate of throughput of powder; at a mean specific powder charge of 1.5 g/cm³ of the hot zone of the reactor the rates of powder throughput for the above-mentioned times of residence are 100-18 g/min. The reducing gas passes through a drying system (a furnace with copper shavings 10 and columns with silica gel 11) and is then fed into the reactor. Its rate of flow is measured with a flow meter 12, and its humidity with a coulometric gas humidity meter. Process temperature measurement and regulation are performed with the aid of a control unit 16.

Final reduction annealing experiments were carried out on PZh5M fine reduced iron powder to GOST 9849-74 (total Fe = 97.4-97.5%, O = 1.2%), covering a wide range of particle sizes, and a powder produced by milling incompletely reduced Sulinsk Metallurgical Plant sponge iron (total Fe = 95.2-95.8%, O = 2.8-3.1%). The oxides present in these materials are mainly in the form of wustite inclusions (primary oxides [9]) in particles. The presence of secondary oxides (Fe₃O₄), in the form of thin surface films, was revealed only by a selective analysis of particles from the outer, "blue" part of the sponge-iron tube.