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EFFECT OF CONDITIONS OF MANUFACTURE ON THE PROPERTIES OF ULTRAFINE NICKEL POWDER

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The steady expansion of the fields of applications of ultrafine powders (UFPs) necessitates studying and developing their methods of manufacture. In particular, there is a need to identify the factors influencing the particle size of UFPs. Of special interest in this connection is the levitation method [1], which enables UFPs to be produced with very narrow particle size ranges and mean particle sizes varying from 0.01 to 0.5 μm in a continuous process. The present work was therefore undertaken with the aim of examining, using nickel UFPs as an example, the effect of the key parameters of production of particles by the levitation method on their size. Use of a ferromagnetic material made it possible to study at the same time the effect of the dimensional factor on the magnetic properties of such UFPs. The first results of this investigation have been reported in [2].

As can be seen from the diagrammatic representation shown in Fig. 1, evaporation of a metal drop 1 in the apparatus employed is performed in a suspended state with the aid of a countercurrent generator 2 supplied from a VChG4-10/0.44 high-frequency generator 3. Nickel vapor entrained by an inert gas stream condenses, with the formation of spherical particles (Fig. 2), which are trapped by a filter 4. At the end of the process the particles are removed from the filter by means of a rod 5 into a capsule 6, another rod 7 serving to withdraw a sample of the particles for electron microscopy. During evaporation fresh metal is continually supplied to the drop from a wire of the starting material 8, which passes through a feeding device 9 and a stuffing box 10. The velocity of the cooling inert gas stream v is regulated by means of a valve 11 and a rotameter 12. In our work all investigations were carried out at an inert gas pressure close to atmospheric. A study was made of the effect of the rate of particle condensation s, rate of flow of the cooling inert gas, and its atomic weight on the size and magnetic properties of the nickel particles being formed. Magnetic properties were studied using a vibration magnetometer.

The rate of condensation for levitation under self-regulating conditions is usually made equal to the rate of consumption of the metal wire [1], which can easily be regulated by varying the speed of rotation of the feeding device rollers. In the apparatus employed the rollers were driven by an RD-09 reversing motor through a special reductor. The linear wire feed rate in the system was varied steplessly and in steps in the range 0.03-30 mm/sec (for 0.2-mm-diameter nickel wire the corresponding variation of s was from 0.01 to 10 mg/sec). At constant rates of flow of inert gas through the vaporizer the dependence of the mean nickel UFP particle size $\bar{d}$, calculated on the basis of analyses of micrographs, on s was as shown in Fig. 3. The power-function character of the $\bar{d}$ vs s relationship was maintained with various inert gases and at various gas flow rates. The slopes of the straight-line plots in the figure indicate a typical $\bar{d} \propto s^{1/2}$ relationship.

In [1] it is noted that $\bar{d}$ decreases with increasing rate of flow of cooling inert gas. In that work the type of this relationship was determined for a constant rate of evaporation of material, the quantity directly recorded being the rate of flow of inert gas Q = const v. As can be seen from Fig. 4, the log $\bar{d}$ vs log Q relationship has a characteristic slope corresponding to $\bar{d} \propto Q^{-1/2}$. According to our data, this formula is valid for the gas flow rate range 10^{-6}-10^{-3} m^3/sec, or the gas velocity range 10-10^4 mm/sec, respectively. The identical character of the $\bar{d}$ vs Q relationship for three inert gases with different atomic weights M made it possible to establish that $\bar{d} \propto M^{1/2}$.

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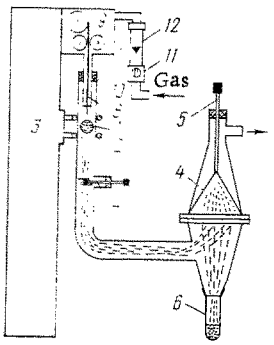

Fig. 1

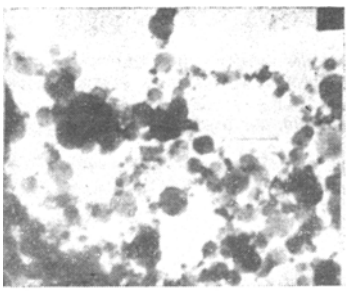

Fig. 2

Fig. 1. Diagrammatic representation of apparatus for production of UFPs.

Fig. 2. Ultrafine nickel powder. Length of gauge area 0.2 μm.

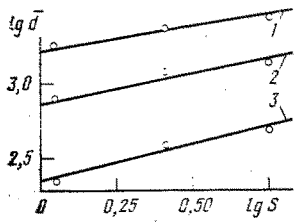

Fig. 3

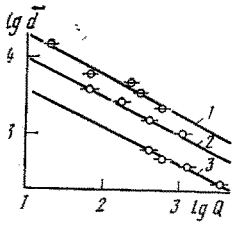

Fig. 4

Fig. 3. Variation of mean UFP particle size with rate of material evaporation at given rates of flow of inert gases — argon (1 and 2) and helium (3). Q = $4 \cdot 10^{-5}$ (1) and $2 \cdot 10^{-4}$ m^3/sec (2 and 3).

Fig. 4. Variation of mean UFP particle size with rate of flow of cooling inert gases — xenon (1), argon (2), and helium (3). Condensation rate 1.1 mg/sec.

Thus, adding together the above $\bar{d}$ vs s, Q, and M functions, for the mean size of metal particles produced by the gas condensation levitation method we can write

$$\bar{d} = k\sqrt{sM/Q}\,. \tag{1}$$

The coefficient k would be expected to depend on such process characteristics as gas pressure, energy expended in vaporization, vapor pressure of the metal, and geometric dimensions of the vaporizer. In principle, all these parameters can be controlled and stabilized. Investigations have shown that one or two experiments are sufficient for calibrating any such apparatus.

It has been noted earlier [1] that increasing the gas flow rate narrows the particle size range and shifts it toward smaller values of $\bar{d}$ according to Eq. (1). In such a case the particle condensation zone apparently becomes elongated in the direction of the inert gas stream [1]. This means that, when a mixture of two inert gases is used, the condensation zone, which determines the mean particle size of a UFP, will presumably increase in proportion to their contributions to the total stream. Our investigations have shown that the mean size of particles produced, e.g., in a mixture of argon and helium, is given by the expression

$$\bar{d}(\mathrm{Ar}+\mathrm{He}) = \frac{\bar{d}_{\mathrm{Ar}} \cdot Q_{\mathrm{Ar}} + \bar{d}_{\mathrm{He}} \cdot Q_{\mathrm{He}}}{Q_{\mathrm{Ar}} + Q_{\mathrm{He}}}\,. \tag{2}$$

It is interesting to note that Eq. (2) can be very helpful in understanding the mechanism of condensation of UFP particles. In fact, when a triangle is used as an approximation to the normal logarithmic particle size distribution [3], Eq. (2) resembles the expression for its

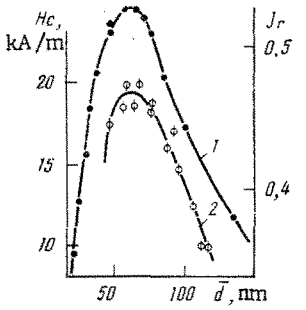

Fig. 5. Variation of H_c (1) and J_r (2) with mean size of particles produced in argon and helium gas mixture.

height. The area of such a triangle is equal simply to the sum of the areas under the distribution of particles in the case of mono gases, with the number of particles being assumed to be directly proportional to their size range. This implies also a direct relationship between the number of evaporated metal atoms (and hence metal particles) and the number of inert gas atoms cooling them. Apart from this, included here is an indication of the energetic character of the condensation zone. It must be added also that use of a gas mixture is very convenient. The point is that with a single gas it is not always possible to ensure a given UFP particle size without the stability of the liquid metallic drop being disturbed by a high-velocity gas stream or without rapid settling of particles on the inside surface of the quartz tube of the vaporizer.

Powders produced in a mixture of argon and helium gas streams were used not only for testing the validity of Eq. (2) but also for determining such magnetic characteristics of nickel particle assemblies as coercive force H_c and relative residual magnetization intensity J_r, which are indicative of the degree of rectangularity of a hysteresis loop. Figure 5 shows the variation of these characteristics with mean particle size. The maximum values of H_c and J_r in this figure apparently correspond to the ranges in which a single-domain state is attained in a nickel UFP, the critical single-domain structure size $\bar{d}_c$ being close to that obtained in [4]. The feasibility of controlling the magnetic parameters of a nickel UFP by varying the composition of a mixture of inert gases provides an interesting means of producing powders suitable for use in advanced data recording systems [5].

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