SINTERING OF LOOSE COBALT, NICKEL, AND IRON POWDERS IN A MAGNETIC FIELD

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Gravitational forces exert an appreciable influence on shrinkage during the sintering of pressed compacts as well as of loose powders [1]. Unlike locally applied external loads, they act simultaneously on all volume elements of a solid. Now the action of magnetic forces on a disperse ferromagnetic powder system is similar in character to that of gravity, and in a magnetic field, too, the magnitude of the force acting on a specimen varies over its height. Unlike that of gravitational forces, however, the direction of magnetic forces can be varied, and apart from this magnetic forces can be much larger than gravitational forces. In view of this, it is of interest to study the effect of a magnetic field on densification and on the formation of properties during the sintering of loose ferromagnetic powders — disperse systems capable of being deformed under the action of light loads. Such an investigation can be of considerable practical importance and at the same time provide a scientific basis for a number of production processes in which use is made of the action of a magnetic field on the sintering of ferromagnetic powders.

In the present work a Weiss–Pogorelyi electromagnet supplied with current from two dc power sources was employed. At a pole shoe diameter of 100 mm and a pole gap of 100 mm the field strength attained ~ 0.3 T. The fine electrolytic cobalt powders and carbonyl nickel and iron powders chosen for investigation were sintered in the as-poured and tapped conditions. Powder samples were placed in a quartz tube through which argon was passed. The quartz tube was surrounded by a nickel–chromium alloy heating element which enabled a temperature of 1473°K to be reached. Electrical and heat insulation was provided by an asbestos sheath and a water-cooled copper tube unit (Fig. 1). The whole system was held between the pole shoes of the electromagnet.

Powder samples were sintered in 8- and 17-mm-diameter, 40- and 20-mm-long capsules. During sintering the capsules were arranged parallel to the magnetic force lines. The metals of the powders studied have the following Curie points: cobalt 1404, iron 1041, and nickel 670°K. The sintering of the powders in a magnetic field was performed at temperatures both below and above the Curie points. For purposes of comparison specimens were also sintered, under the same conditions and at the same temperatures, in the absence

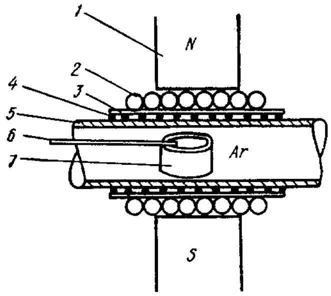

Fig. 1. Diagrammatic arrangement of setup for sintering in magnetic field: 1) electromagnetic pole shoe; 2) water-cooled copper tube unit; 3) asbestos lining; 4) electric heating element; 5) quartz tube; 6) thermocouple; 7) container with powder.

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TABLE 1

Rel. height of horizontal specimen section	H, T	F, N
0,1	0,221	6,5
0,3	0,206	3,4
0,5	0,193	2,25
0,7	0,183	0,96
0,9	0,175	0,45

TABLE 2

Powder	Sintering conditions	Magnetic field	$\rho_{por} \cdot 10^7$, $\Omega \cdot m$
	As-poured		
Co_{el}	1393 K; 1800 sec	Absent	240
	The same	Present	68
	Tapped		
	1393 K; 1800 sec	Absent	90
	The same	Present to 673°K	60
	» »	Present	45
Fe_{carb}	973 K; 1800 sec	Absent	125
	The same	Present	72
Ni_{carb}	1393 K; 1800 sec	Absent	52
	The same	Present to 673°K	46
	» »	Present	49

of a magnetic field. To sensure that a consolidating magnetic force was generated, during sintering current was passed through the winding of only one pole shoe.

After sintering the specimens were subjected to porosity and volume shrinkage determinations involving hydrostatic weighing. To assess the degree of perfection of interparticle contacts, which is a criterion of quality of sintering, measurements were made of the electrical resistivities of specimens and, using the method described in [2], of the rates of propagation of ultrasonic vibrations through them in a direction parallel to the magnetic field lines.

The mean magnitude of the magnetic forces acting on a powder system in a magnetic field was found by determining the force necessary to pull a capsule with the cobalt powder in a tapped condition off a pole shoe at room temperature (a 10-g powder sample in a 17-mm-diameter, 20-mm-high capsule was used). The distance from the specimen to the magnetic pole was the same as during the sintering of specimens. The force was found to be equal to 6.5 N. As the magnetic forces acting in horizontal sections of a specimen were greatest at its base (lying near the surface of the pole shoe) and least at its upper face, determinations were made of the magnetic force gradient over the specimen height. The magnetic force diminished with increasing height owing to decrease in the volume of the ferromagnetic (in the same way the gravitational force decreases with increasing height owing to decrease in the mass of material) and with increasing width of the gap between the shoe and specimen section under consideration.

As magnetic field strength is an exponential function of distance, this relationship can be expressed with the formula [3]

$$\overline{F} = \mu_0 \varkappa_0 \cdot c \cdot V \cdot H_0^2 \cdot e^{-2cr}, \tag{1}$$

where $\overline{F}$ is the ponderomotive force; μ_0, magnetic permeability of vacuum; χ_0, magnetic susceptibility of a particle; V, volume of the ferromagnetic; H_0, maximum magnetic field strength; r, size of the gap, and c, a geometric constant.

The variation of the magnetic field strength as a function of distance from the pole surface was determined by experiment, using a teslameter, and the variation of the magnetic force over the specimen height was calculated, assuming the force at the specimen base to be equal to the force necessary to pull off a specimen (6.5 N) (Table 1). From the magnetic force gradient obtained we find that the mean value of the force acting on the specimen was ≈ 2.7 N. This force corresponds to a mean effective external specimen of ~ $1.2 \cdot 10^4$ Pa.

To study the effect of a magnetic field on densification during sintering, experiments were conducted with the cobalt powder because of its high Curie point. A sample of the cobalt powder in a tapped condition (starting porosity 70%) in a quartz capsule was sintered for 1800 sec in an argon atmosphere of 1073°K. At this temperature the relative saturation magnetization intensity of cobalt amounted ~ 80%. The volume shrinkage of the cobalt powder was found to be equal to 13% after sintering in a magnetic field and 5.5% after sintering without a magnetic field. These values correspond to specimen porosities of 65.5% after magnetic sinter-

TABLE 3

Powder	Sintering conditions	Magnetic field	USV speed, m/sec
Co_{el}	1393 K; 1800 sec	Absent	1300
	The same	Present to 673°K	1600
	» »	Present	1900
Ni_{carb}	1393 K; 1800 sec	Absent	3900
	The same	Present to 673°K	4050
	» »	Present	4000

Note: The USV speeds have been reduced to the same porosity.

and 68.2% after conventional sintering. Thus, the application to a disperse ferromagnetic system of volume magnetic forces substantially surpassing volume gravitational forces has a marked intensifying effect on powder densification during sintering.

The operation of magnetic forces during sintering results also in changes in the physical properties of sintered specimens. Electrical resistivity measurements were made on cobalt, nickel, and iron powder specimens sintered with and without a magnetic field in 8-mm-diameter, 40-mm-long quartz capsules (Table 2). The electrical resistivity of cobalt powder specimens sintered in a magnetic field was only one-third to one-half that recorded after ordinary sintering. These results demonstrate that sintering in a magnetic field ensures a much higher degree of perfection of interparticle contacts in specimens.

The reason why the effect of a magnetic field on sintering quality is greater with an as-poured than with a tapped powder is that a disperse system in the as-poured condition experiences greater deformation under the action of a light load. When a magnetic field was present only up to 673°K, its effect on the electrical resistivity of cobalt specimens was much less marked. The same field strongly affected also the quality of sintering of tapped iron powder specimens in experiments in which the sintering temperature did not exceed the Curie point. The influence exerted by a magnetic field on the sintering of the nickel powder was vary slight because a magnetic field acts on nickel at temperature below 671°K, i.e., temperatures at which the processes of viscous flow of metal at low stresses are severely impeded.

The rates of propagation of ultrasonic vibrations (USVs) through specimens sintered from tapped cobalt and nickel powders with and without a magnetic field are presented in Table 3. The data of this table bear out the results of electrical resistivity measurements, and are evidence that a magnetic field substantially increases the degree of perfection of interparticle contacts in the sintering of a cobalt powder, but has only a slight effect on the sintering of a nickel powder.

In connection with what has been said above, it is interesting to note that, with what appears to be only a small difference in porosity between cobalt specimens sintered in a magnetic field and by the orthodox method, the difference in physical properties between these specimens is very substantial. It may be that this apparent discrepancy is linked with some of the sintering characteristics of loose powders described in [4]. In particular, it is shown in that work that in the sintering of an unpressed powder the size of interparticle contacts (which controls the level of physical properties) is directly determined by the volume densification of the powder, and an expression is proposed relating the relative linear size of contacts to the change in porosity brought about by sintering,

$$\xi^2 = 1 - \left(\frac{\theta}{\theta_0}\right)^{4/3}, \qquad (2)$$

where θ_0 is the starting porosity and θ is the porosity after sintering.

In [4, 5] a formula is given for calculating the electrical resistivity of a porous solid with imperfect interparticle contacts,

$$\rho_{por} = \frac{\rho_0}{\xi(1-\theta)^{3/2}}, \qquad (3)$$

where ξ is the relative linear size of an interparticle contact, ρ_{por} and ρ_0 are the porosities of the porous and nonporous materials, respectively, and θ is the porosity. In the present work we calculated, using Eq. (3), values of ρ_{por} for tapped cobalt powder specimens sintered in a magnetic field and without it. Relative contact diameters for substitution in Eq. (3) were determined with Eq. (2).

For specimens sintered in a magnetic field and by the conventional technique values of ρ_{por} of $118 \cdot 10^{-7}$ and $210 \cdot 10^{-7}\ \Omega \cdot m$, respectively, were obtained. Thus, when the porosity of specimens sintered in a tapped condition changes from 68.2 to 65.5%, their electrical resistivity can be expected to fall by almost one-half; this was in fact confirmed by electrical conductivity measurements on such specimens. These results indicate that the same laws govern the sintering of unpressed powders in a magnetic field and without it. Consequently, the growth of interparticle contacts in both cases is linked directly with the volume densification of porous material.

The marked improvement in physical properties (measured in the direction of action of the magnetic force lines) induced by magnetic sintering can probably be attributed also to powder in a magnetic field being arranged anisotropically as a result of particle reorientation, but this hypothesis requires further investigation.

CONCLUSIONS

The action of volume magnetic forces markedly surpassing volume gravitational forces on a disperse ferromagnetic system strongly intensifies the densification of the powder during sintering. The substantial improvement in physical properties exhibited by porous specimens after magnetic sintering indicates a higher degree of perfection of interparticle contacts, resulting from the intensification of densification achieved during sintering in a magnetic field, and, possibly, an anisotropic arrangement of powder in a magnetic field.

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