Jan. 9, 1940. H. VOGT 2,186,659

MAGNETIC POWDER FOR IRON DUST CORES

Filed July 6, 1937

Fig. 1

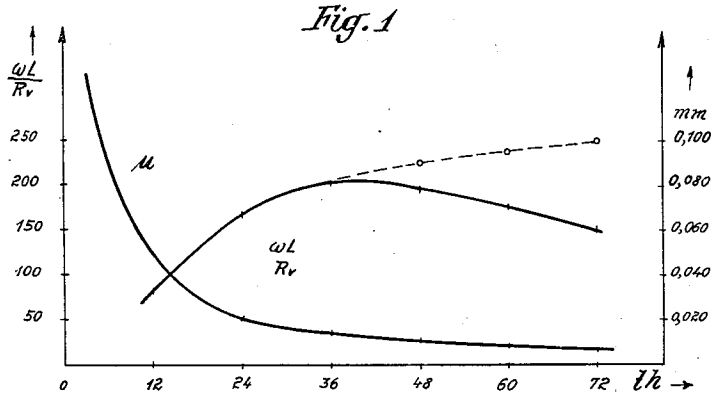
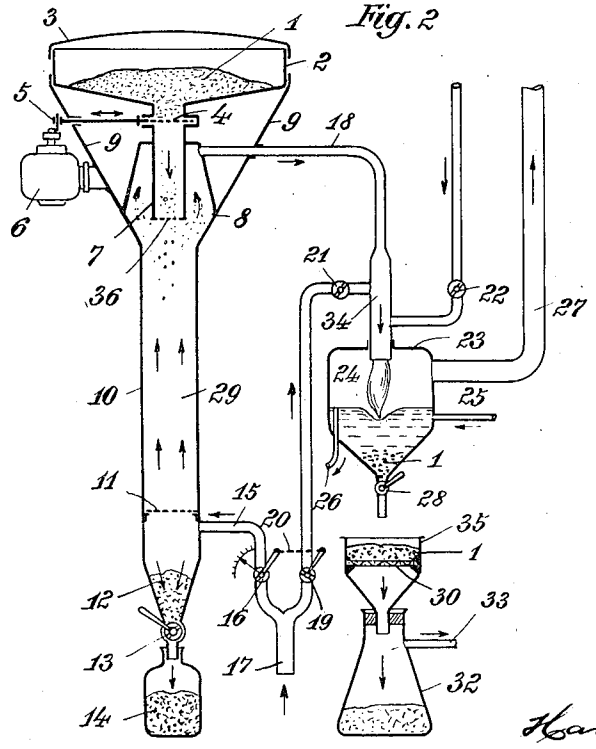


Fig. 2



Inventor:
Hans Vogt,
By Potter, Pierce & Schaffler
Attorneys.

Patented Jan. 9, 1940 2,186,659

UNITED STATES PATENT OFFICE

2,186,659

MAGNETIC POWDER FOR IRON DUST CORES

Hans Vogt, Berlin-Steglitz, Germany, assignor to Micro Products Corporation, Hastings-on-Hudson, N. Y., a corporation of Delaware

Application July 6, 1937, Serial No. 152,204
In Germany July 17, 1936

6 Claims. (Cl. 148—1)

The present invention relates to a method for the production of magnetic powder which may be suitably compressed to nuclear masses with an insulatory agglomerant having minimum loss at frequencies of about 1000 kilocycles, perferably without setting up an organic insulating film. The purpose of this method is to produce in a specially economic manner this kind of magnetic powder and to obtain the finest quality as regards high-frequency losses and mechanical firmness at the same time. According to the invention the powder is produced from finely divided alloy consisting of iron with silicon and/or equivalents which increase the brittleness, hardness and the specific resistance, as e. g. aluminum, in particles of dimensions below 0.02, preferably even less than 0.01 mm. The particles are melted to balls by blowing through a compressed gas flame and collected in a manner that the minuteness of the particles and their spherical form are preserved.

The powdered alloy can be produced chemically or mechanically to the required fineness and composition. The chemical production can take place, starting with iron as a salt or in the form of an oxide, e. g. $Fe,(OH)_2$, which is either first reduced and then mixed, or first mixed with the silicon in powder form and then reduced.

During the heating of the mixture to about 500°–700° C. in the presence of hydrogen, the iron is reduced at the same time as the silicon diffuses into the iron. A sintering of the powder does not take place in this process. One can also commence with ground iron powder and alloy it with the silicon as in the above described manner.

In the mechanical comminution of the powder transformer sheet-iron may be utilized, having a silicon content up to 4% and which can be suitably given a preliminary grinding to the required fineness in a propeller mill. Especially advantageous is the use of silicon alloys having a silicon content above the ductility limit and yet below the range of non-magnetic alloys. This corresponds to a silicon content of about 5–15%. Ferric silicide produce in a blast furnace is most suitable and is best cooled off as quickly as possible after melting, e. g. by leading it over a rotating plate or through a screen and cooling it off with water. By this means the hardness and brittleness of the granulated metal are raised considerably and the comminution facilitated. A typical analysis of an original material thus produced is the following:

	Per cent
Iron	82.07
Carbon	1.74
Silicon	11.4
Manganese	0.29
Phosphorus	0.652
Sulphur	0.008
Aluminium	1.59
Titanium	2.25

The impurities contained therein are not only non-injurious, but they actually facilitate pulverization. Methods for the production of this kind of powder by comminution have already been described in applicant's copending application Serial Number 61,112, filed January 27, 1936.

Pulverization takes place effectively by wet or preferably dry mechanical grinding in two runs of a ball mill. In the first run comparatively coarse balls are applied and in the second run much smaller ones. The burden of the mill can be increased by forcing a stream of gas through the mechanism and thus the sufficiently small ground particles are continuously sifted through the mill. In this manner, the full crushing power of the balls acts upon the particles that are still too large and the cushion effect of the smaller particles is eliminated. In order to avoid undesired reactions (e. g. oxidation) the gas stream can at the same time serve as a protective gas and contains for this purpose hydrogen, nitrogen, illuminating gas, carbonic acid or similar substances. The product coming from the mill is then balled in the compressed gas flame and the balled material is caught in a liquid bath.

By a proper selection of the feed-powder as well as the air-gas proportion, speed and direction, a suitable speed can be imparted to the particles so that they do not coalesce during flight and the temperature of combustion is increased to such a degree that the particles are melted in the course of their rapid motion. The flame contains an excess of reducing gases by which the danger of oxidation of the particles is lessened. After melting, the powder can be

retained in a vessel heated to 700–900° for a certain time whereby the loss of permeability caused by cooling of the powder is compensated. The ball-shaped powder can, in addition, be winnowed by air.

The material thus produced is then immediately mixed with the agglomerating agent preferably consisting of artificial resins which can be solidified in such a manner that at least 30% of the volume of the agglomerating agent, preferably in dissolved form, are mixed, i. e. kneaded with the magnetic powder. The compression is then performed in the well-known manner.

The utilization of mechanically ground powder with high silicon content is already known in the Pupin nuclear coils. But such powders were not suitable for the special requirements in high frequency nuclei since they resulted in too high losses on account of their coarse granulation and irregular shape. The applicant has already suggested the utilization of mechanically ground ferric silicide for high frequency purposes. This suggestion did not, however, bring about any successful result because the particles were mechanically rounded. It was found that this mechanical method was insufficient for the extremely small-sized particles required in high frequency nuclei. It has already been proposed to produce magnetic powder for mass nuclei by reducing iron wire to dust by means of a metal spraying device and conducting it into a cooling medium. In thus reducing the metal there are produced particles of a size which although entirely sufficient for use with Pupin coils are, far too coarse for high-frequency nuclei, causing great loss.

The special advantages derived from the utilization of ferric silicide rounded in the flame were not recognized in this suggestion regarding high-frequency nuclei. The method proposed for the making of glass balls by introducing pulverized material into the flame could not offer any suggestion for the solution of the present special problem. The manner in which the powder is introduced into the flame outside the burner employed in this method does not permit as even a distribution as is necessary in the present case.

It had also to be assumed that the extremely fine iron particles, necessary for high-frequency iron nuclei, would burn up or at least oxidize to a great degree, or in other respects be affected disadvantageously resulting in a very inferior powder. This opinion is expressed in former works of the applicant in which he suggested that the substance be led through a chamber which is heated externally so as to avoid the direct effect of the flame. But it became evident that in applying this procedure to industrial mass production, the small particles conglommerated on a large scale, thus giving rise to numerous coarse particles. On the other hand, by merely subjecting the substance to treatment by direct flame, hitherto considered impracticable for such fine powders, balling without agglomeration of the particles is achieved. In all former instructions for thermic balling, knowledge was lacking concerning the true significance of the silicon content in the attaining of the desired result.

Only through the utilization of a substantial silicon content and the brittleness attained thereby is it possible to comminute the particles to a sufficiently fine state by means of mechanical grinding. The fineness of the powder thus obtained gives ample insurance against any loss. The suitable range of the size of the particles is not increased and the thermic balling improves the quality of the product on account of the higher specific resistance caused at the same time by the high silicon content.

Characteristically, all reference as to procedure for the balling of the particles is lacking, especially in the proposals to utilize alloys of supraductility which have a high silicon content in the preparation of nuclear masses. For it was believed that in view of the high internal resistance the increased insulation of the particles, obtainable with particles of round form, was superfluous. This is, in fact, true to a certain extent for with the progressive pulverization to about 20μ, I get first a certain optimum in regard to the proposed losses. The particles are of completely irregular form and even balling does not result in any improvement.

When the pulverization is continued the powder becomes finer though it becomes electrically inferior. But even the best powder thus obtained is not of the same value as carbonyl iron, which, up to the present, has produced the best nuclei. Inasmuch, then, as the use of even high-silicon-content alloys and the balling of the particles best obtained thereby, has not resulted in the production of a powder equivalent to carbonyl iron, this method had to be disregarded. In view of the high requirements in the selectivity of the receivers which decidedly depend on the degree of loss of the high-frequency iron nuclei, only the nucleus with minimum loss can be considered in the greatest number of cases applied. Strangely enough, it was found that the passing of the at first inferior, finer powder through a blow-flame does not only make the latter equal in value to the coarser powder, but may even be enhanced in value and become equal in quality to carbonyl iron or even superior to it. Such improvement could not in any way be expected.

The invention thus rested on the realization that it was possible by means of a definite combination of operations to produce a high-frequency pulverized iron, equal in value at least electrically to carbonyl iron powder, until then resulting in least loss and which is considerably superior to it economically. The advantage derived from my powder consists especially in the possibility of utilizing cheaper parent material together with a simple method of manufacture. But the powder thus produced, owing to its high internal and surface resistance and because of its greater hardness, may be continuously compressed merely by the application of a sufficient volume of binding media without the additional formation of an organic insulating film on the individual particles.

The omission of the insulation process means a considerable simplification of manufacture, as well as the elimination of sources of error. Moreover, the nuclei thus produced are especially firm and mechanically durable. The procedures used in combination are the displacement of iron with a high silicon content or an equivalent metal (e. g. aluminium) in the form of particles of magnitude less than 0.02 mm. and the thermal balling of the powder thus attainable in the blow-flame. After mixing the powder with a sufficiently large quantity of binding agent, nuclei are obtained showing a minimum loss at a frequency of more than 1000 Khz. At this stage, it is necessary to understand that such a nucleus of small loss is one which brings about a considerably greater increase in self-induction when introduced into a

high-frequency coil than the increase in resistance.

I shall now describe the invention in greater detail with reference to the accompanying drawing:

Figure 1 is a diagram in which we can see the connection between time of pulverization, magnitude of particles, form and quality.

Fig. 2, a plant for the sifting and thermic balling of the powder.

In Figure 1 the longer of the two curves which slopes entirely downwardly to the right shows the maximum magnitude of the particles in millimeters indicated by the letters mm. and plotted as ordinates, in relation to the time of pulverization indicated by the letters *lh* and plotted as abscissa. The other curve gives the corresponding values of a coil indicated by the symbol

$$\frac{\omega L}{Rv}$$

and plotted as ordinates with a core produced from a powder the particle size of which is plotted as abscissa in terms of the pulverization time corresponding to the particle size. Thus it is evident that a particle size of about 0.015 mm. gives the optimum coil value and that with continued pulverization the coil value not only does not improve, but actually becomes inferior (solid line curve). However, if the particles thus pulverized are led through a blow-flame, we get a sudden increase in coil value, but only in particles of a size less than 0.020 mm. (dotted curve). The increased value thus gained, results in reference to the prepared self-induction coils, in those very values (which are a measure for the value of the coil and therefore for the selectivity of the receivers) which make possible the general application of the nuclei thus produced even demanding the highest quality.

Fig. 2 shows a suitable plant for sifting and thermal balling. The parent powder to be used can be iron powder either mechanically pulverized or chemically prepared.

According to the arrangement in Fig. 2 the iron powder 1 is poured into the feeding-funnel 2, supported by means of braces 9, and closed with the cover 3. The powder drops through a shaking sieve 4, driven by motor 6, by means of the eccentrics 5 into the induction tube 7, the bottom of which can be closed by a coarse sieve 36. The size of this sieve 4 and the manner in which shaking takes place regulates the amount of powder passing through the contrivance in a given time. An air-current 29 flows up through tube 10 colliding with the powder which is dropping from tube 7. The powder which is of sufficient fineness is forced by the current of air into tube 18 by way of the annular canal formed at 8.

The annular cross-section at 8 should not measure more than the internal cross-sections of tube 10, so that no greater velocity of gas results than in tube 10. By this means, the dragging-along of the larger, i. e. too heavy particles is prevented. The larger, heavier particles fall down rather perpendicularly through tube 10 and through the coarse sieve 11 and collect at 12. From here the particles are emptied into a receptacle 14 by means of a drawing-off mechanism and can be used again in the mill for further comminution. The air current 29 is regulated by means of a gauge 16 in the air-pressure tube 17—15. The regulatory gauge 16 can be connected with gauge 19 by means of a coupling rod 20 or similar positive device in such a manner as to result in the same total volume of flow in any region between the two gauges. In this way, the power of the air-current 10, determining the maximal size of the particles, can be regulated by valve 16, without affecting the total volume of air forced through the burner.

The air current flowing through gauge 19 can be additionally throttled at 21 for the purpose of regulating the total volume of air. Then within tube 34, it mingles with the air current carrying the powder flowing through channel 18 and also the fuel gas passing through the regulator 22. The flame 24 is so formed that in a short time it melts the conducted particles into balls. The flame 24 strikes against the water surface found in receptor 23 which is kept at a constant level by means of the feed-pipe 25 and overflow tube 26.

The powder 1 is immediately cooled off in this manner and is collected in the lower part of the receptacle 23, from which it can be drawn off by means of the stopcock 28. The powder is thus drawn off into the suction filter below, consisting of receptacle 35, together with filter screen 30 and collecting receptor 32. The moisture adhering to the powder flows through the filter screen and collects in receptor 32, during which process powder 1 can from time to time be removed from the upper part of receptacle 35. By means of the tube 33, the air below the filter can be withdrawn through suction (vacuum) which hastens the drying process.

The receiving fluid in receptacle 23 may be water to which preferably alkaline ingredients are added. I prefer a receiving fluid having a pH-value of about 9 to 10; 8 is the lower and 11 the upper limit in most practical cases. The alkaline character may be produced by adding ammonia, soda lye and/or potash lye to the liquid. I contemplate, moreover, to add to the alkaline receiving fluid ingredients acting as reducing agents in alkaline solution. One or more of the known photographic developing substances may be used for this purpose, such as for instance rodinal, pyrogallol, methol and/or hydrochinon and/or formalin. Stabilizators, such as for example sodium carbonate and/or ammonium carbonate may be added to the reducing agent in order to avoid premature neutralization of the reducing agent.

The powder can now be subjected to an annealing-treatment for 12–48 hours in vacuo or in nitrogen at temperatures of 700–900° C. which improves its permeability.

The powder thus prepared can then be mixed in a kneading machine with the binding medium, preferably artificial resin that can be solidified amounting to more than 30% by volume, or in dissolved form. After drying the substance is ready to be compressed into high-frequency nuclei of the required shape and in accordance with the well-known method.

The balling process described in this invention, according to which the powder to be balled is first sifted by means of a gas current and then blown and melted in a compressed gas burner by means of the sifting gas current, may also be successfully applied to similar fusible powdered substances. The special method of feeding the powder to the burner by means of the sifting gas current renders possible a very even distribution of the powder. In this way, agglomeration and feeding of quantities that are too large are avoid-

ed for the burner cannot melt such masses at a sufficient rate.

I claim:

1. In a process of producing magnetic powder, the steps which comprise producing a powdered alloy consisting of iron and about 5% to 15% of silicon, mechanically disintegrating said alloy to magnetic particles of a size less than 0.02 mm., blowing said magnetic particles through a reducing gas flame at a temperature sufficient to melt the particles to spherical form, and cooling down and collecting the particles so obtained.

2. A process as claimed in claim 1, characterized in that the particles are collected after melting in a receiving fluid consisting essentially of water.

3. A process as claimed in claim 1, characterized in that the particles are collected after melting in a receiving fluid of alkaline nature.

4. A process as claimed in claim 1, characterized in that the particles are collected after melting in a receiving fluid of alkaline nature, containing ingredients which act as reducing agents in alkaline solution.

5. A process as claimed in claim 1, characterized in that the particles are collected after melting in a receiving fluid of alkaline nature, containing ingredients which act as reducing agents in alkaline solution and stabilizators for said reducing agents.

6. In a process of producing magnetic powder, the steps which comprise blowing magnetic particles of a size less than 0.02 mm. through the flame of a gas burner by means of a compressed gas current to which the magnetic powder has been added, said flame being at a temperature sufficient to melt the particles to spherical form, leading to said burner a parallel gas current which carries no magnetic powder, the proportion of the two gas currents being adjustable while maintaining the gas quantity of the total gas currents constant, and cooling down and collecting the particles so obtained.

HANS VOGT.