페로코발트 자성유체의 제조와 응용

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Preparation and Application of Ferro-Cobalt Magnetic Fluid

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1. Introduction

Magnetic fluid contains powder of strong magnetic materials, such as magnetite, iron and nickel suspended in liquid, and the powder is not separated with the application of centrifugal force or a magnet. An early implementation of magnetic fluid was the fuel supply system in an aerospace ship. Since a wide variety of applications were developed, mass production of the powder has been investigated. A mass production technique was developed by Sato and Shimoiizaka[1] using a chemical process.

One of promising applications of the magnetic fluid is fluid sealing from a rotating shaft. An iron particle dispersed magnetic fluid was utilized in the sealing of a high rotation pump[2], and it is found that the sealing holds up to the pressure of 618 kPa with a rotating shaft of 1800 rpm. A similar application to a rotary blood pump having rotation of 10,000 rpm was also reported[3]. A slightly different implementation of the magnetic fluid has been conducted in the development of a safety valve[4], which has a mechanical spring to hold the pressure. The magnetic fluid replaces the spring with its magnetic force.

A mechanical seal used for preventing fluid leakage from rotating pumps requires holding pressure of 2.5 kg/cm^2, but usual magnetite magnetic fluid has only 1 kg/cm^2 of holding pressure. In this study, a ferro-cobalt magnetic powder containing 30 % of cobalt is manufactured for the implementation in a high holding pressure seal. The powder is coated with silicon dioxide for the prevention of aggregation and surface active agents for the dispersion in fluid medium. The size and property of the powder are investigated, and its sealing performance is experimentally tested.

2. Experimental

2.1 Materials and Instruments

As raw materials of ferro-cobalt powder, waste pickling liquor from steel cleaning process, cobalt chloride (Junsei Chemical Co., Japan) and nitric acid (Matsuen Chemical Co., Japan) were used. Hydrogen (Hana Specialty Gas Co., Korea) for reducing agent and sodium silicate (Ilsin Industries, Korea) for powder coating were also used. The powder was dispersed in ethylene glycol (Junsei Chemical Co., Japan) to compose a magnetic fluid. Surface active agents of oleic acid (Hayashi Pure Chemical Co., Japan), dodecyl benzene sulfonate (D. B. S., Hayashi Pure Chemical Co., Japan) and tetramethyl ammonium (T. M. A., Acros Organics, U. S. A.) were applied for the stable dispersion of the powder. Lubricating oil (SAE 5W/40, Honam Refining Co., Korea) was used in the sealing performance test.

The particle observation of the powder was conducted with an X-ray refractometer (Model D/MAX-II, Rigaku Instruments, Japan), and particle size and shape were determined with a scanning electron microscope (SEM, Model S-4200, Hitachi Instruments, Japan). In the process of sample preparation for SEM measurement, the powder was coated with gold in the thickness of 18 nm and the thickness is deduced from the actual measurement. Magnetic property of the sample was measured with a vibrating sample magnetometer (Model 7300, Lakeshore Instruments, U. S. A.). Acid durability of the coated powder was determined with a thermal analyzer (Model TG/DTA32, Seiko Instruments, Japan) for the temperature from ambient to 200 °C at the temperature increase rate of 10 °C per minute.

2.2 Experimetnal procedure

Waste pickling liquor from steel processing is used as a source of iron to manufacture ferro-cobalt powder. The liquor is utilized here for waste recycling, and its composition is listed in Table 1. Nitric acid are added to the acid in the concentration of 1 % for the oxidation of Fe^{2+} ion to Fe^{3+} ion. The actual oxidation occurs from blowing air into the solution. Then, cobalt chloride is put into the solution to formulate the ratio of 7 to 3 of iron and cobalt. In the mixture, caustic soda is added to make hydroxide precipitation in a strong basic solution of pH of 12. The reaction occurred at room temperature for 6 hours. The precipitation is separated with decantation, and washed with water more than 10 times. After the precipitation is filtered with 5A filter paper and dried at the temperature of 60 ± 5 °C, sodium silicate is applied for the coating of the powder. The detail of the coating procedure is as below.

The dried powder 20 g is put in 1 L of distilled water, and the silicate is added to maintain the content of 3.1 to 4.8 wt % ratio of SiO_2/Fe-Co with continuous agitation. At room temperature, the solution is agitated for 2 hours. The coated powder is filtered and dried at 100 °C. The cobalt ferrite powder is reduced with hydrogen in a horizontal tube furnace at the temperature of 500 °C for 6 hours. The flow rate of the hydrogen is 0.5 L per minute.

In order to give hydrophilic property to the powder, the powder is placed in 0.1 M oleic acid solution for 1 hour with agitation. Again, the treated powder is washed with water 8 times and filtered. It is dried in an oven at the temperature of 60 ± 5 °C. The treatments with D. B. S. in the concentration of 0.1 M and T. M. A. in 0.01 M give more hydrophilic property. Finally, the powder is dispersed in ethylene glycol to compose a magnetic fluid. The sealing performance of the magnetic fluid is examined by applying it to the sealing mechanism as shown in Figure 1.

3. Results and Discussion

3.1 Property of the powder

The ferro-cobalt powder obtained in this study has particle size distribution of 3 to 29 nm, and average size for 50 % cumulative value is 9.4 nm which is small enough to have Brownian motion when dispersed in liquid. The measured magnetization of the powder is 154 emu/g in the magnetic field of 10 kOe. It is 64 % of the theoretical value of 240 emu/g. The magnetization is maintained up to the temperature of 150 °C.

In order to prevent from aggregation among the ferro-cobalt particles, the particle surface is coated with silicon dioxide and surface active agents for the even dispersion in liquid. Also the liquid should not be mixed with oil that is the material to be sealed and should not evaporate to air. In this study, ethylene glycol is utilized because it is hydrophilic and has high boiling point.

For the examination of settling property of the powder, two kinds of powders with different coatings of surface active agents are mixed with ethylene glycol in the concentration of 61 % and agitated with a shaker for 2 hours. The mixture is placed in a measuring cylinder to determine the settling height with elapsed time as demonstrated in Figure 2. Figure 3 shows the height variation for 20 weeks. While the powder of three coatings with oleic acid, D. B. S. and T. M. A. gives little variation, the powder having two coating of oleic acid and D. B. S. shows significant settlement. Fujita, et al. [5] explained that the triple coating is composed of oleic acid in the innermost, D. B. S. in the next and T. M. A in the outermost. The thickness of the double coating of oleic acid and D. B. S. is reported as 4.9 nm and the triple coating is 10 nm. It is known that these thick coating helps the dispersion of the powder due to the repulsion among the coated surface.

The relation between magnetization and Fe-Co powder content in the magnetic fluid is illustrated in Figure 4. As the content is raised, the magnetization increases until the content of 70 % in which the fluid loses fluidity and becomes gel. Magnetization property of the Fe-Co magnetic fluid is investigated with a magnetization curve indicated in Figure 5. The magnetic fluid of powder content of

15 % is placed in various strength of magnetic field to yield the curve. Though the powder is a strong magnetite, no hysteresis is observed in the property measurement.

3.2 Sealing application

In order to examine oil sealing performance of the Fe-Co magnetic fluid, an experimental setup given in Figure 6 is prepared. A motor with power of 1 hp (Dongyang Motors Co., Korea) equipped with a variable speed transmission is connected with a cylinder holding lubrication oil. The motor shaft is attached to the rotating shaft in the oil cylinder through the sealing mechanism described in Figure 1. The cylinder has two seals at the both ends and the shaft is installed through the cylinder. Two pole pieces and a permanent magnet are implemented in the mechanism. The gap between the rotating shaft and the pole piece is 0.5 mm. The magnet is an Fe-Nd permanent magnet having surface magnetization of 3200 Gauss, and its dimension is 48 mm in O. D., 33 mm in I. D. and 7mm in thickness. The pole piece is made of 1 mm thick pure steel.

A 20 mL of magnetic fluid is injected in the sealing mechanism of the experimental setup. Also the oil holding cylinder is filled with 150 mL of lubrication oil. While the shaft is turning at 1800 rpm, nitrogen gas is supplied to increase the cylinder pressure. When a magnetic fluid of 70 % content is utilized, the outcome of performance test is shown in Figure 7. In order to elevate the holding pressure, multiple sets of sealing mechanisms are installed in parallel. As the number of the mechanism increases, the holding pressure becomes higher. In the comparison with a conventional magnetite magnetic fluid, the Fe-Co magnetic fluid exhibits 25 times higher holding performance.

4. Conclusion

Ferro-cobalt powder is manufactured to develop a magnetic fluid seal used in the sealing of highly pressured oil. The powder is coated with silicon dioxide for the prevention of aggregation and three different surface active agents for a good suspension of the powder in sealing fluid media, ethylene glycol.

The particle size of the powder is 9.4 nm in average, and little settlement of the particle is observed after 20 weeks settlement test in liquid dispersion. The particle magnetization is 154 emu/g in the magnetic field of 10 kOe without hysteresis. The sealing performance test indicates that the ferro-cobalt magnetic fluid has 25 times higher holding compared with a conventional magnetite magnetic fluid.

References

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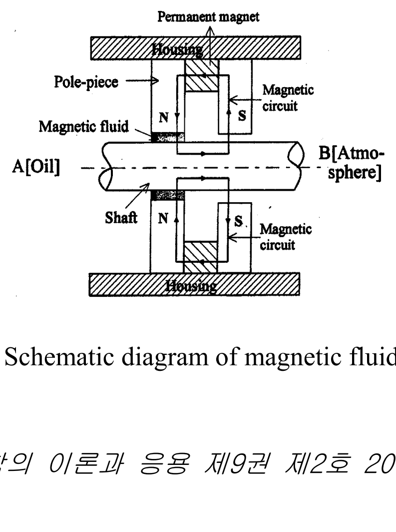

Figure 1. Schematic diagram of magnetic fluid seal mechanism.

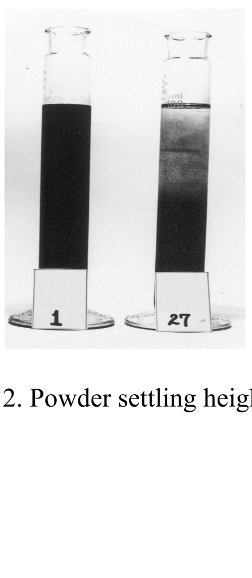

Figure 2. Powder settling heights.

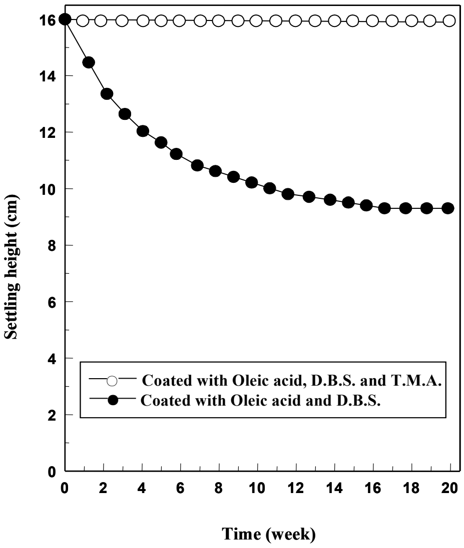

Figure 3. Settling of magnetic powder coated with two different combinations of surface active agents.

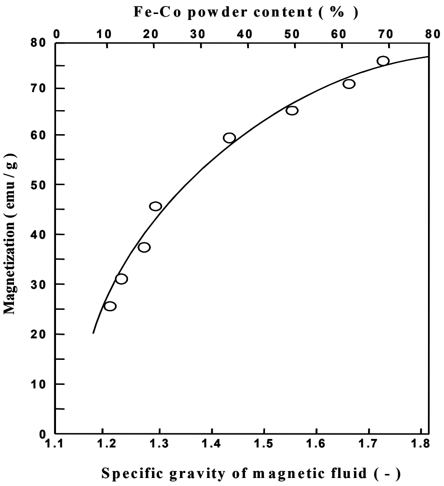

Figure 4. Magnetization strength with different contents of Fe-Co powder

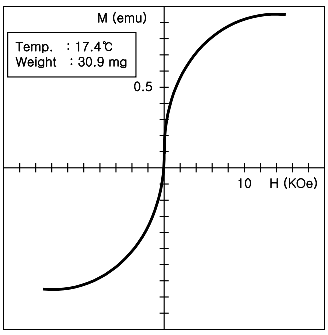

Figure 5. Magnetization curve of silica coated Fe-Co magnetic fluid.

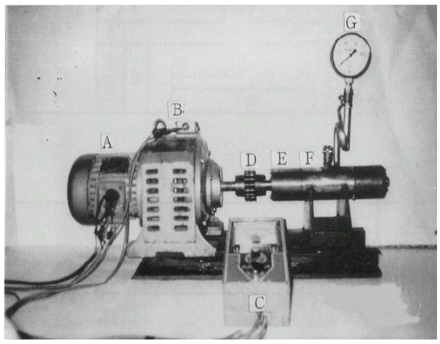

Figure 6. Photograph of experimental setup. (A: motor, B: variable speed transmission, C: speed controller, D: coupling, E: sealing mechanism, F: lubrication oil cylinder, G: pressure gauge)

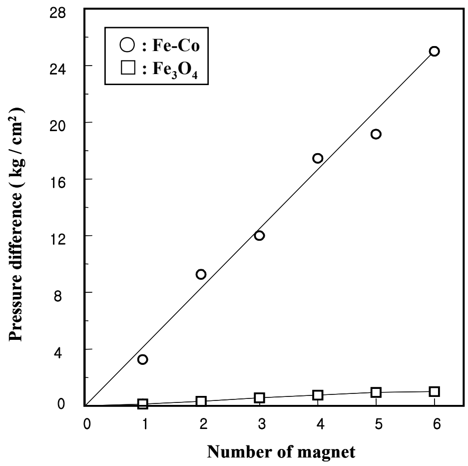

Figure 7. Relation of magnet number and holding pressure for two different magnetic fluids.

Table 1. Chemical composition of waste pickling liquor from steel treatment. (units in wt. %)

component	content
Fe^{2+}	8.69
Fe^{3+}	0.26
Mn	0.03
Cr	0.005
Cu	0.0005
Zn	0.001