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Development of ferrofluid and its possible applications in MEMS

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In micro-fluidic devices, fluid control plays a crucial role in its operation. The input and output in the device must pass through control surface. Synthesized nano magnetic fluid is characterized by X-ray diffraction (XRD), transmission electron microscope (TEM) and vibrating sample magnetometer (VSM) techniques for its potential applications in MEMS devices as gas pressure valve. Operation of gas pressure valve was monitored at varying electromagnetic actuation level.

Keywords: Ferrofluid, MEMS, Micro-valve, Crystallite size

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1 Introduction

Micro-fluidic chambers are the fundamental components in a variety of micro-devices such as labs-on-chips, micro-sensors and micro-total analysis system[1,2]. In the realm of nano tech materials, ferro-fluids are unique in their constituents in both liquids and solids. Ferrofluids are the colloidal dispersion of nano size 2-10 nm magnetic particles in some suitable carrier medium and have varieties of applications in micro-devices, like in Robotics, micro valve, biomedicine, phase shifters, composites and sensors etc[3-5]. Ferrofluids potentially provide very good seal and response to external localized magnetic forces, providing easy actuation. This makes them advantageous over other methods of fluid control. The present investigation deals with the preparation of $CoFe_2O_4$ nanocrystalline particles by chemical co-precipitation method and its utilization in micro valve. In a nano phase material, particle size, shape, crystallinity and their distribution always play a crucial role in fabricating devices. Therefore, these magnetic particles were characterized by x-ray diffraction (XRD), transmission electron microscope (TEM) and vibrating sample magnetometer (VSM) techniques. Under the application of external magnetic field, an additional body force $M\nabla(H)$ is generated in a ferrofluid, where M is the magnetization of the fluid and $\nabla(H)$ is the field gradient.

These fluids are very stable against gravitational and magnetic forces and magnetic in nature, so their flow can be controlled by the application of magnetic field. A micro valve has been designed by utilizing these properties to control the gas/liquid flow. In the present work, we machined a micro valve inside silicon single crystal with anodically bonded Pyrex glass. The device has been designed and developed to control the gas flow at micro level by magnetic actuations. The potentiality of the valve was measured at varying actuations levels. This investigation focuses on magnetically and electro magnetically actuated control in order to find its use for the different pressures in MEMS devices.

2 Theory

In a ferrofluid seal, the burst pressure of the ferrofluid valve is obtained with the help of well-known ferrohydrodynamics Bernoulli's equation. The burst gas pressure that a ferrofluid plug in a channel can withstand is obtained with good approximation by:

$$\Delta P = \mu_0 \int_0^{H_{max}} M dH = \mu_0 \overline{M} H \quad \ldots \quad (1)$$

where $\overline{M}$ is field average magnetisation;

$$\overline{M} = \frac{1}{H} \int_0^{H_{max}} M dH$$

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To calculate this pressure, one needs to know the maximum field H_{max} at the position of the valve and is given by ($H_{max.} = B_{max.}/\mu_0$). Ferrofluid plug is generated by the permanent magnet and the relationship given in Eq. (1) was tested quantitatively[6].

3 Experimental Details

The material was synthesized by chemical co-precipitation method using 1 molar aqueous solutions of high purity salts of Co $(NO_3)_2.6H_2O$ and $Fe(NO_3)_3$. $9H_2O$. These solutions were mixed in 1:2 ratios and homogenized at constant temperature (70°C). Thereafter, with continuous stirring *p*H of the solution was maintained at 10.5 by adding 25% ammonia solution. The material, thus, obtained was used for the ferrofluid preparation[7]. Material was characterized by XRD, VSM and TEM techniques for its crystallinity, magnetization and particles shape and size distribution.

The development of a micro fluidic-valve is based on a ferro-fluidic actuation. Here ferrofluid has been actuated to block a channel with a pressure difference of maximum 0.095 atmospheres that could be used for the flow of a gas. In the present work, a cylindrical gas channel of diameter 0.5 mm in single crystal silicon has been designed. It is fabricated using bulk micromachining[8] of (100) single crystal silicon. At the center of the channel, a V-shaped groove was formed to accommodate the ferrofluid injected through a micro-syringe. The silicon wafer with the channel and the V-groove is anodically bonded[6] with a 7740 Pyrex glass with an inlet and outlet, as shown in Fig. 1 (a and b).

To actuate the ferrofluid, a series induction is best planned under the channel. The micro-valve can be switched on/off by changing the current in the coil. Under the influence of varying magnetic field inside solenoid, the magnet and ferrofluid valve position is changed within the silicon micro-channel to monitor the gas pressure. Moving the ferrofluid back into the V-groove by the action of magnetic field through inductor will open and close the micro-valve. The total device is of the size 10 × 10 × 15 mm^3 with inlets and outlets as shown in Fig. 1 (a & b).

Also, we have studied the pressure difference, which the valve is able to hold on varying the magnetic field strength at valve position. A schematic diagram of valve with different magnetic field strength is shown in Fig. 2 (a and b).

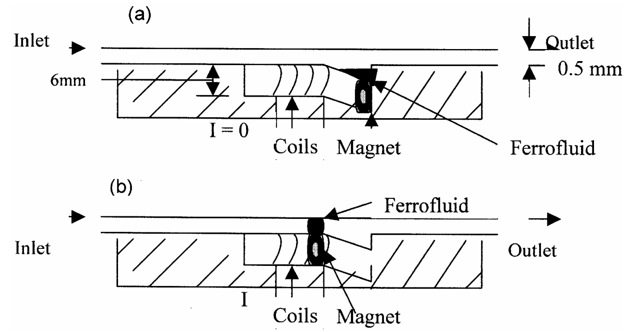

Fig. 1—(a) & (b) Change in ferrofluid valve position with variation in coil current.

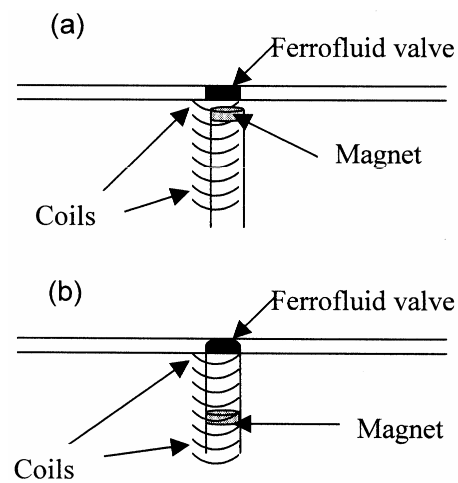

Fig. 2—Variation in valve shape and strength with distance from the magnet (a & b)

4 Results and Discussion

In a nano phase material particle size, shape, crystallinity, magnetization and their distribution always play a crucial role in fabricating devices. The crystalline phase analysis was determined by D-8 advance powder X-ray diffractometer. The XRD pattern of the sample shows a single crystalline phase of $CoFe_2O_4$ where intensity and *d* values of all the observed diffraction peaks match with the cubic form of cobalt ferrite (ICDD data file No-22-1086) as shown in Fig. 3. The crystallite size was calculated by using the Scherrer formula. A slow scan of (311), (400), (511) and (440) diffraction peaks was done to calculate the full width at half maximum (FWHM). The crystallite size calculated by using Scherrer formula[9,10] is of the order of 6 nm. There are various techniques to determine the particle size distribution. Here, we have used TEM measurement technique to determine the shape and size distribution of the particles. Fig. 4 (a) shows the representative electron

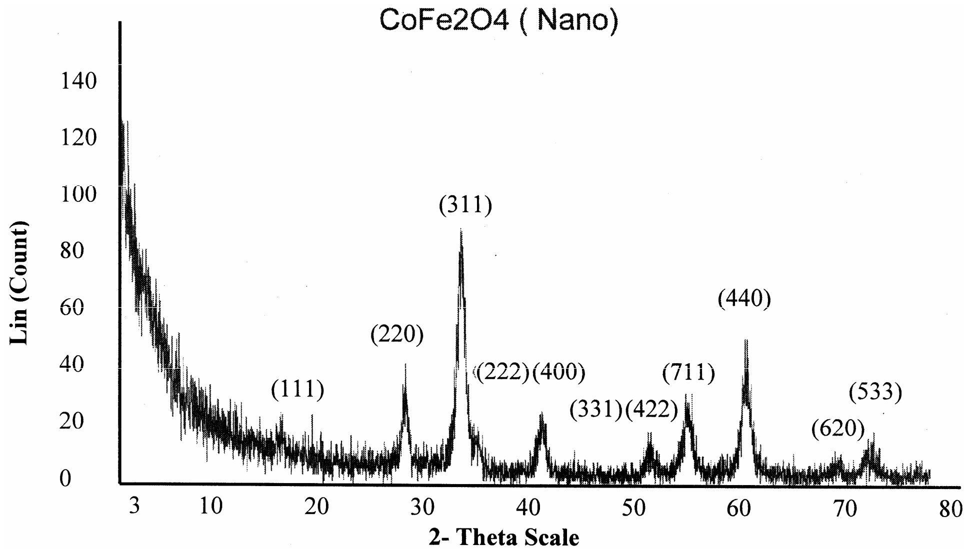

Fig. 3—XRD pattern of the nano crystalline magnetic particles of $CoFe_2O_4$

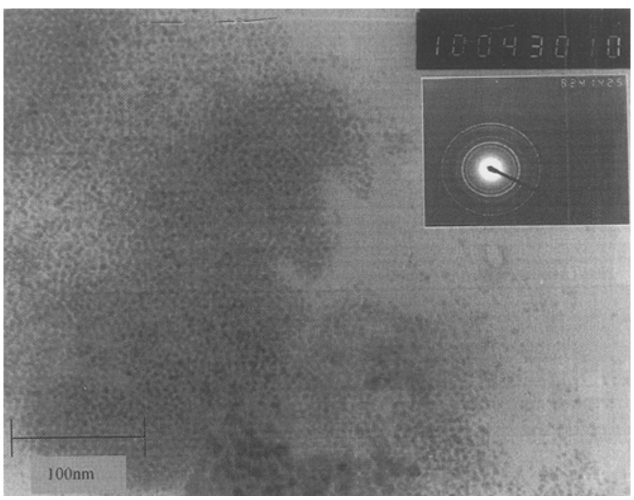

Fig. 4 (a)—TEM Micrograph of $CoFe_2O_4$ particles with inset showing the corresponding electron diffraction

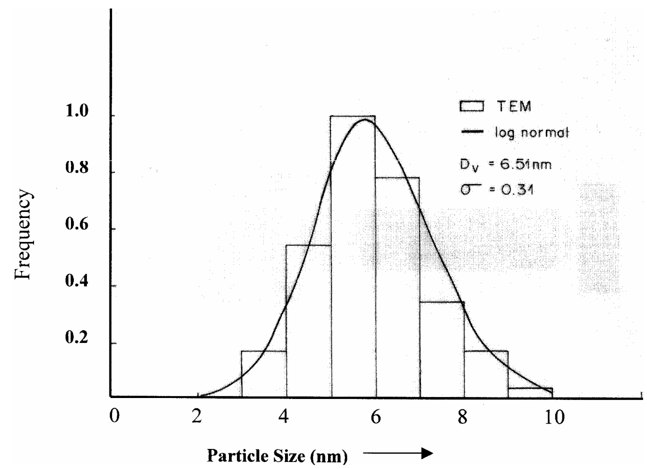

Fig. 4 (b)—Relevant particle size histogram showing the size distribution

micrograph and inset reflects the electron diffraction rings. The inter planer spacing calculated from the diameter of the diffraction rings perfectly match with the crystalline phase of cobalt ferrite. It is seen that almost all the particles are spherical in shape and have uniform size distribution in the range 5-10 nm.

Fig. 4(b) shows the particle size histogram obtained from the as synthesized cobalt ferrite TEM micrograph. The curved line represents the best fit of particle size using the standard log normal distribution function having $D_v = 6.51$ nm and $\sigma = 0.31$.

The results of studies carried on magnetic moment versus applied magnetic field (5×10^3 Oe) at room temperature of the sample using VSM are shown in Fig. 5. The VSM curve of the as-prepared sample shows the superparamagnetic behaviour of the particles with unsaturated magnetization 55 emu/gm and the data shows that majority of particles are superparamagnetic (SP) in nature. SP size behaviour is observed when the thermal energy kT of the particles exceeds over the magnetic energy KV. This is in good agreement for using this fluid for the MEMS applications. The saturation magnetization (Ms) of the fluid is 200 G.

Micro-valve is one of the most promising microflow control devices[11]. We have designed a ferrofluid based micro-valve in which micro actuation is electrically controlled. Nearly 6 μL ferrofluid was

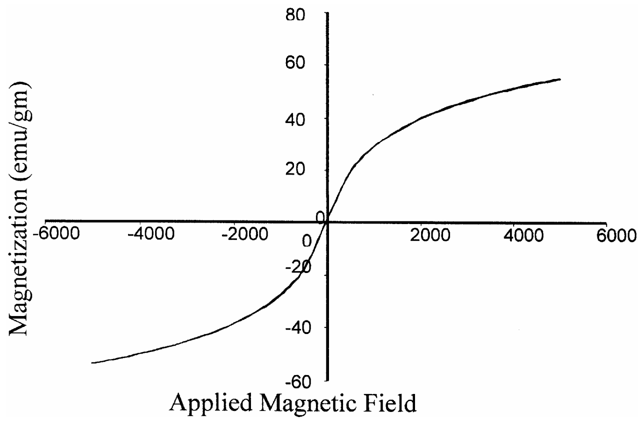

Fig. 5—*M-H* curve showing the room temperature magnetization of cobalt ferrite Nano-particles

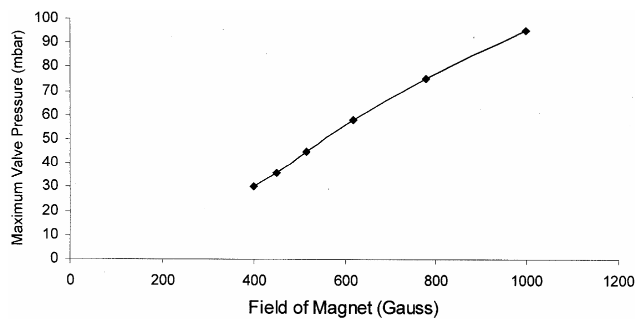

Fig. 6—Graph showing the maximum pressure valve can hold as a function of magnetic field at valve position

placed inside the channel, which makes a valve of cylindrical dimensions of 0.25 × 5 mm. Using NdFeB (N50) permanent magnets (diameter 5 × 3 mm, Br = 1400 mT, coercive field *H*c = 840 kA/m) with ferrofluid bearing which is actuated by the copper coil in turn actuates the valve. The magnetic fluid was introduced in the channel with a syringe and placed at the correct initial position with the external magnet situated over the channel. The device is able to control the rate of gas flow at a pressure difference of 95 mbar. The performance of the micro-valve mainly depends on its working principal. At micro level, this type of valve or seal provides contamination free gas flow. The ferrofluid valve allows the continuous flow of gas from input to the output reservoir. The valve is active type, which normally opens in case there is no current in the solenoid. On increasing the current through the solenoid, the electromagnetic actuation of ferrofluid valve starts responding. By further increasing the actuation current from 0-25mA, the valve can efficiently withstand a pressure difference (Δp) of 95 mbar as presented in Table 1. Beyond this value, the applied pressure exerts enough force to displace or burst the fluid. Rate of flow of gas was monitored by counting the bubbles at outlet by varying the actuations level as shown in Fig. 6. The valve strength in the varying induction level in the range 400-1000 G has been studied. Table 2 presents the detail of the operation of the valve at different values of magnetic field at the valve position. At 400 G the micro-valve can hold a maximum pressure difference of 30 mbar while it can efficiently withstand a pressure difference 95 mbar at 1000 G. This way the valve can operate at micro pressure range. Thus, the performance of micro-valve seems to be well suited for the Micro Electromechanical System. The micro fabrication of biocompatible material is an excellent technique for disposable biomedical devices and very low in cost.

Table 1—Performance level of electro-magnetically actuated ferrofluid valve

S.No.	Actuating Current mA	Δ_P mbar
1	0.00	0.00
2	10.00	20.00
3	15.00	40.00
4	20.00	65.00
5	25.00	95.00

Table 2—Variation of valve strength as a function of magnetic field at valve position

Sr. No.	Distance of magnet from micro-channel (mm)	Magnetic field at valve position (Gauss)	Maximum pressure valve can hold (mbar)
1	0	1000	95
2	2	780	75
3	4	620	58
4	6	516	45
5	8	452	36
6	10	400	30

5 Conclusions

Ferrofluid microvalve actuation is controlled by electrically induced magnetic field. In the realm of miniaturization of technology, this type of valve may find good applications in many new generation devices like in micropums to control the gas pressure at micro level, targeted drug delivery and in future, it will be interesting to realize micro-valve with improved control.

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