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Characterization of Low Pressure DMA System for the size selection of magnetic nano-particles

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ABSTRACT For the size selection of magnetic nano particles produced in laser ablation, a low pressure differential mobility analyzer (LPDMA) was constructed. The LPDMA was characterized using the transmission electron microscopy (TEM) image of laser-ablated Co-Pt nano particles. Using TEM image, the geometrical standard deviation, σ_g, was measured to be around 1.13. The dependence of the performance of LPDMA on the gas path temperature and the pressure was measured using an electrode which measures the number of the selected particles.

The size-selected magnetic nano-particles were deposited on Si substrates, whose magnetizations were measured by SQUID. It was found that nano-particles with a diameter of 20 nm have different temperature dependences from nano-particles with a diameter of 40 nm and the coercivity of 40 nm dia. nano-particles is smaller than that of 20 nm dia. nano-particles.

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1 Introduction

Due to its size, the magnetic property of a nano-particle has different characteristics from bulk. Special magnetic characteristics can be found from a nano-particle because the size of the particle can be smaller than that of a single magnetic domain. The dependence of magnetic properties of a nano-particle on its size have been widely studied [1].

Pulsed laser ablation is a simple method to make nano-particles. However, the size distribution of particles produced in laser ablation is broad. A size selection apparatus is needed to make a sample of mono-dispersed nano-particles. In this work, a low pressure differential mobility analyzer (LPDMA) [2] was constructed and used to select the size of Co-Pt nano-particles produced in laser-ablation.

2 Experiment

The experimental system consists of a laser ablation chamber, a furnace, a differential mobility analyzer, and a deposition chamber, as shown in Fig. 1. To evaporate particles from a target, a Q-switched Nd : YAG laser was used. The pulse duration of the laser was about 5.4 ns with a repetition rate of 20 Hz, and with a power of 800 mW. The target was a mixture of Co and Pt, with Pt being 52.5% (mass weight ratio). To prevent the shot-to-shot variation of plume due to the damage of the target, the target was rotated at a speed of 8 rpm. He gas was used to carry particles to the DMA. The flow rate (0.4 l/min) of He gas and pressure of the DMA were controlled by mass flow controllers and variable conductance valves. He gas carried nano-particles through a furnace. The furnace was used to reduce the particle agglomeration [3]. The temperature of the furnace was varied from room temperature up to 800 °C.

The low pressure differential mobility analyzer (LPDMA) system was used to measure the particle distribution and to select a particular size of particles [2] after the furnace. The ability of a particle in gas to move in an electric field is expressed in terms of the particle's electric mobility which is given by the terminal velocity of the particle in gas divided by the applied electric field. The electric mobility depends on the inverse of the particle size. If the gas which contains the

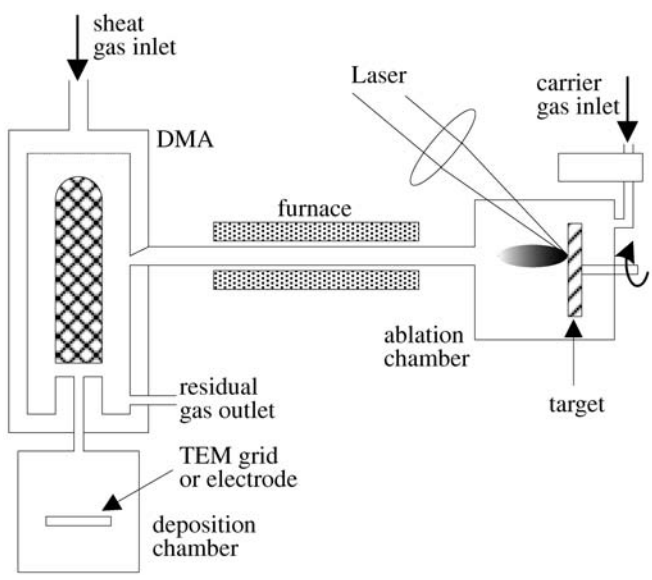

FIGURE 1 Schematic diagram of experimental setup

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particle flows downward as in Fig. 1 and the electric field is applied between the center rod and outer well, the position where the particle meets the center rod is determined by the electric mobility. Particles with different sizes meet different positions of the center rod [4]. An output aperture placed at the center rod extracts only particles with a particular size. By varying the voltage across the center rod and the outer well, the size selection can be achieved. The diameter of the center rod of the DMA was 22 mm and the inner diameter of the outer cylindrical shell was 36 mm. A 10 mm-long center rod was used for the measurement of small-size particles and a 210 mm-long rod for the measurement of large-size particles. To transport particles in a vertical direction in the DMA, He gas at a flow rate of 2.0 l/min was used as a sheath gas. Using mass flow controllers both at the carrier gas inlet and at the deposition chamber, the flow rate of the carrier gas and selected particle flow was matched.

A deposition chamber was attached to the DMA outlet. In the deposition chamber, the electrode with a electrometer was used to measure the number of selected particles by measuring the current due to charged particles from the DMA outlet. The measurement of the current from the electrode with respect to the dc voltage applied to DMA yields the size distribution. In the measurement of the size distribution, TEM micro-grids were used to collected particles in the deposition chamber.

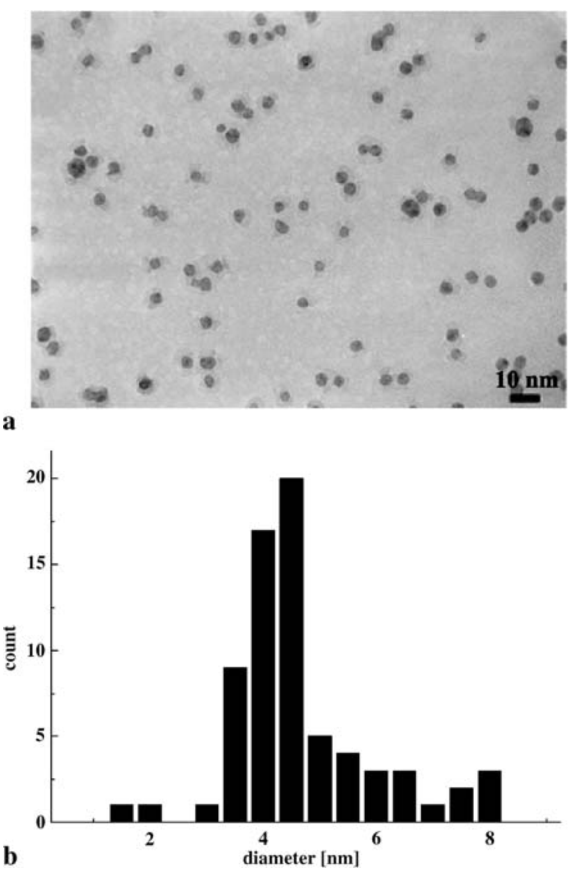

FIGURE 2 Size distribution of selected particle by LPDMA. **a** TEM image of selected particle; **b** the particle size distribution measured from **a**. The voltage of DMA was decided by selecting the 5 nm diameter particle. The geometrical standard deviation was 1.13

3 Results and discussion

Using TEM, the size distribution of the DMA with respect to an applied voltage was characterized. The pressure in the DMA was 9.0 Torr and the temperature at the furnace was 800 °C. The geometrical standard deviation σ_g, which was calculated statistically from the size histogram of TEM pictures, was about 1.13 for a 5 nm-dia. sample and 1.10 for a 10 nm-dia. sample as shown in Fig. 2.

The dependence of the size distribution on the temperature and pressure of DMA was measured using an electrode. Figure 3a shows the size distribution of particles at a pressure of 14.6 Torr. When the pressure and the temperature was changed, the size distribution also changed. Figure 3b shows the geometrical standard deviation, σ_g, and count median diameter (CMD) at room temperature with respect to the pressure of DMA. When the pressure of DMA increased, σ_g and CMD also increased, which means that larger particles are selected when the pressure increases. Figure 3c shows σ_g and CMD at a pressure of 14.6 Torr with respect to the temperature of the furnace. When the temperature increased, σ_g and CMD decreased. This may be due to the increase of

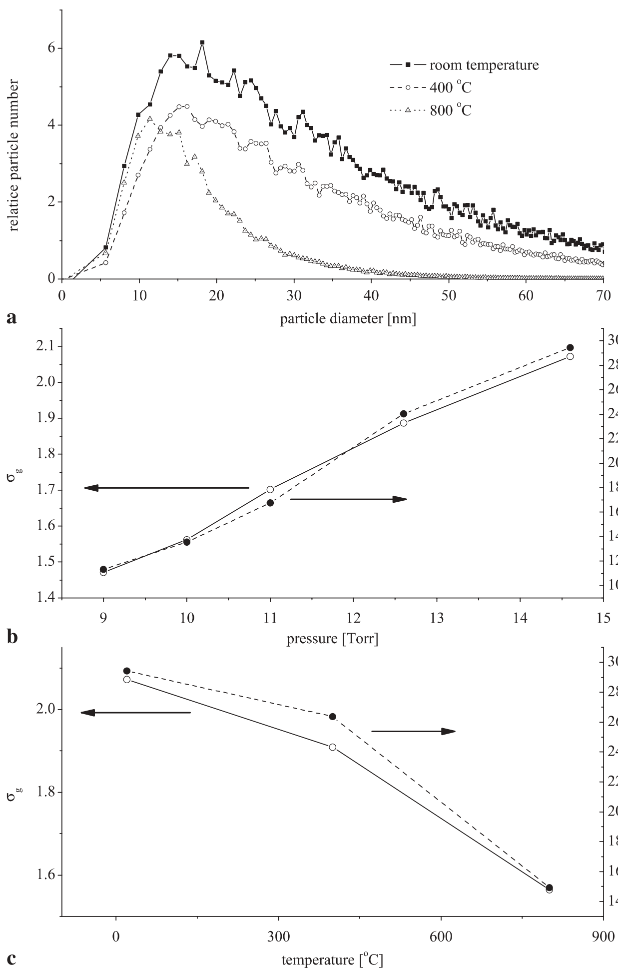

FIGURE 3 Size distribution of nano-particles from laser ablation. **a** is the size distribution with a pressure of 14.6 Torr in DMA and at room temperature. The geometrical standard deviation and the count median diameter measured at room temperature as a function of DMA pressure is in **b** and as a function of temperature at a pressure of 14.6 Torr is in **c**

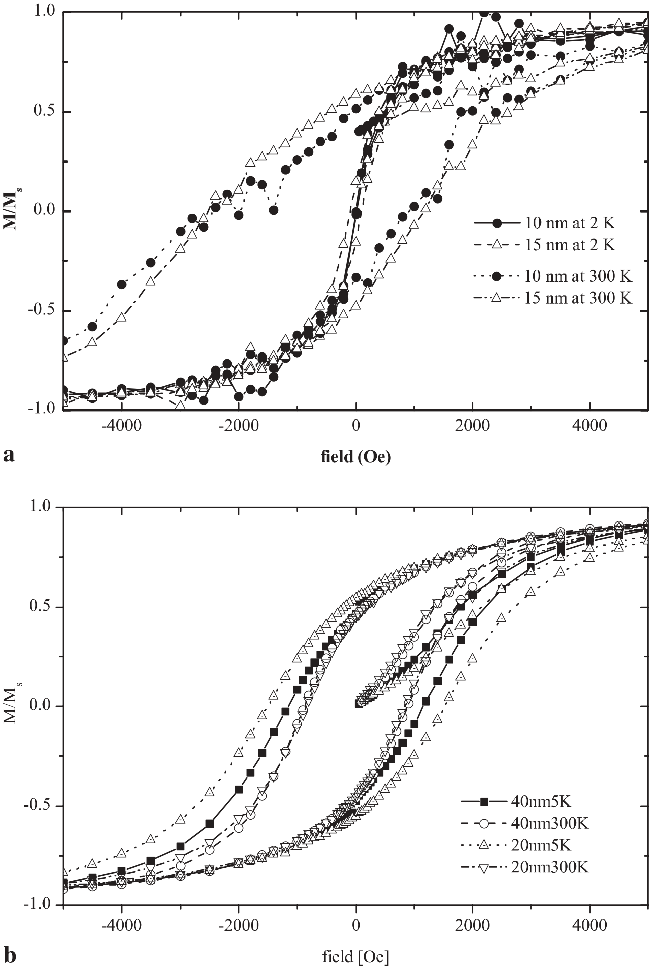

FIGURE 4 Magnetization measurement by SQUID. **a** is for 10 nm and 15 nm size particles measured at 2 K and 300 K, **b** for 20 nm and 40 nm size particles measured at 5 K and 300 K

the mean free path of particles when the temperature was increased [5].

The size-selected magnetic nano-particles were deposited on Si substrates, whose magnetizations were measured by SQUID. Several interesting features were noticed in Fig. 4: the samples of nano-particles with different sizes have different temperature dependencies. Due to the asymmetric magnetization curve for 10 nm and 15 nm diameter particles measured at 2 K, the coercivity was taken as an average value of magnetic fields at which the magnetizations are zero. For this case, coercivities are similar for 10 nm and 15 nm diameter particles as show in Fig. 4a. The coercivity is about 1700 ± 100 Oe. But for the case of 20 nm and 40 nm dia. particles, the coercivity of the sample of 40 nm dia. particles is about 1100 Oe and 20 nm diameter about 1500 Oe when measured at 5 K in Fig. 4b, indicating that the coercivity of the sample of large size particles is smaller than that of small size particles. But at 300 K, the coercivity becomes larger when the particle size is increased: about 0 Oe for 10 nm, 100 Oe for 15 nm, 850 Oe for 20 nm, 900 Oe for 40 nm.

4 Conclusion

To select the size of nano particles produced in laser ablation, a low pressure differential mobility analyzer (LPDMA) was constructed. A frequency-doubled Nd/YAG laser was used to produce nano-particles from a solid target in He gas environment. The TEM image revealed that the geometrical standard deviation, σ_g, was 1.13. The dependence of the particle distribution on the temperature of the carrier path and the pressure of DMA was investigated. When the pressure of DMA was decreasing and the temperature was increased, the particle size distribution became narrower.

The size-selected magnetic nano-particles were deposited on Si substrates, whose magnetizations were measured by SQUID. It was observed that the coercivity of the sample of large size particles is smaller than that of small size particles at low temperature.

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