Metal-Oxide Nanostructure Synthesis

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Introduction

The synthesis of magnetite, Fe_3O_4, nanowires can provide many benefits for society. For example, magnetite nanowires could be employed to improve MRI imaging and communication speed or as a tool for cancer therapy. They are formed by a vapor-liquid-solid (VLS) growth mechanism. It is believed that the nanowire's diameter depends on the size of the deposited Au nanocluster on the collection substrate.[1] Also, hematite, Fe_2O_3, growth is sensitive to the gas flow direction, but this effect on Fe_3O_4 is not known.[2] The goal of this project is to determine the synthesis conditions necessary to permit the growth of aligned magnetite nanowires.

Procedure

Laser ablation was used to grow iron oxide nanowires, but there are three steps in this process. First, a target of Fe_3O_4 is prepared by hydraulic pressing Fe_3O_4 (98% purity) powder into a 3 cm tall by 1 cm diameter cylinder. Second, collection substrates are made by cutting the substrate material with a diamond blade saw. The strips are attached to a silicon wafer using carbon tape, and then placed in a thermal evaporator where 2 nm of gold are deposited onto them. The substrates are detached from the wafer, and the carbon tape residue is removed with ethanol. Third, the synthesis equipment must be prepared. A quartz tube is aligned in the furnace, and a quartz boat is used to hold the target and collection substrates. Then, the boat is moved to the exact middle of the tube to allow the thermocouple to accurately measure the temperature. Using a vacuum pump, the tube is pumped below 17 mtorr. The laser beam is aligned to strike the middle of the Fe_3O_4 target using a lens system. Argon gas is flowed into the tube at a rate of 50 sccm, and the temperature of the furnace is set. The furnace heats the tube at a rate of 20°C/min. Next, the pressure inside the tube is set by adjusting the pump valves. Once the temperature and pressure stabilize to the required synthesis conditions, the Lambda Physik Compex 102 laser (248 nm λ) is turned on for 1 hour and operated at 20 Hz and 30 kV. After cool down, the target and collection substrates are attached to SEM mounts to permit SEM micrographs and XRD analysis to be performed. These analysis techniques were used to view growth alignment and chemical composition.

Results and Discussion

At 7.5 torr, iron oxide nanowire growth occurs between 750 and 950°C. XRD analysis revealed that the majority of the iron oxide formed was hematite and not magnetite. The alignment of the iron oxide nanowires was observed through TFE-SEM. On the polycrystalline Al_2O_3 substrate (Figure 1a), there is nanowire alignment. However, the direction of alignment changes from grain to grain. On the p-type Si substrate (Figure 1b), there is scant growth and it is randomly aligned. The most uniform, complete alignment occurred on the Sapphire substrate (Figure 1c), which is single crystal Al_2O_3. This could allow the Au nanoclusters to all align in the same direction permitting aligned nanowire growth. Also, almost no growth occurred on angled Al_2O_3 (Figure 1d).

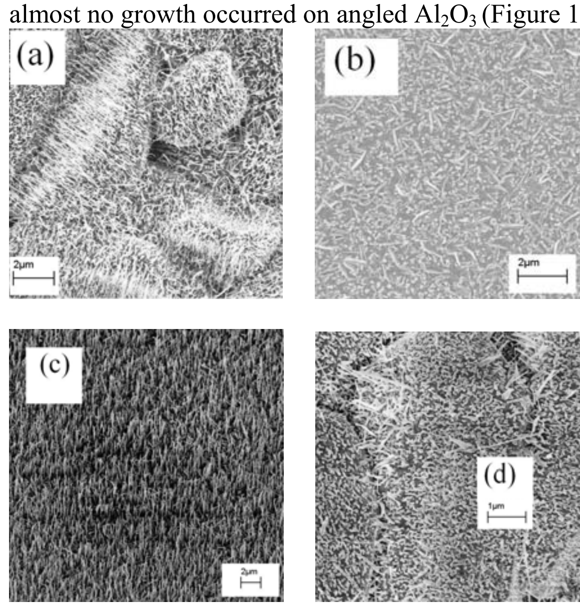

Figure 1. SEM images of iron oxide nanowires grown on varying substrates: (a) polycrystalline Al_2O_3 (950°C, 7.6 torr); (b) p-type Si (100) plane (750°C, 7.4 torr); (c) sapphire (a-plane) (900°C, 7.4 torr); (d) angled 30° polycrystalline Al_2O_3(750°C, 7.6 torr).

Conclusion

Iron oxide nanowires can be synthesized using laser ablation; however, the temperature range for growth is dependant upon pressure, and it is optimal at 7.5 torr when it ranges from 750 to 950°C. Sapphire allowed near uniform, one directional, growth alignment although the majority of iron oxide formed was hematite. Also, gas flow direction did not appear to have any effect other than inhibiting nanowire growth. Furthermore, heat treatment, reduction, and higher purity (99.997%) target experiments are currently ongoing to attempt to convert the hematite nanowires to magnetite.

References

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2. Fu, Y.Y.; Wang, R.M.; Xu, J.; Chen, J.; Yan, Y.; Narlikar, A.V.; Zhang, H. *Chem. Phys. Lett.* 2003, 379, 373-379.