FINAL REPORT (DURIP 00) Scanning Electron Microscope (SEM)

AFOSR Award# F49620-00-1-0261 UD Account# 3-3-21-2570-03

April 1, 2000 – March 31, 2001

George C. Hadjipanayis Principal Investigator

Submitted to: Dr. Harold Weinstock AFOSR/NE 801 N. Randolph Street Room 732 Arlington, VA 22203-1977 (703) 696-8572 20011003 114

The purchase of a high power field emission Scanning Electron Microscope under DURIP FY2000 grant F49620-00-1-0261 has enhanced the Magnetics lab microanalysis capabilities. The Department of Physics at the University of Delaware purchased a state-of-the-art JEOL JSM 6335F Scanning Electron Microscope in the fall of 2000. The purchase price of \$235,000.00 was negotiated to include a high resolution Backscatter detector, motorized X and Y stage control, and infrared specimen chamber scope. The installation was completed in a timely manner and the instrument was turned over to the Magnetics Lab in November 2000.

The increased analytical capabilities of the Magnetics Lab, since the purchase of the field emission SEM, has made the observation of nanostructures in hard and soft magnetic material routine. Having a high power field emission SEM means the laborious sample preparation and extensive time required for TEM imaging is no longer required. Typically, the resolution of the SEM (1.5nm at 15KV) and its depth of field and microanalysis capabilities provide detailed information of samples that previously required sending them to various other facilities. One of the unique features of the field emission SEM is the ability to view elemental maps using EDS techniques. The relatively small beam size provides the resolution to map microstructures and to identify inhomogenities in samples in the range of a few nanometers. Since the installation of the JSM 6335F, our research activities have increased. Quick screening of samples and there associated microstructure can now be performed in a timely manner.

Actual JSM6335F images



Porous Gold Microstructure



Silver Nanoparticles in Polystyrene

One of the more recent undertakings of the UD magnetics lab has been the production of nanoparticles in a matrix using a novel sputtering based deposition technique. Shown below are SEM images of Fe nanoparticles in a BN matrix. Images at 30k magnification give particle sizes in the range of 20~100nm. At higher magnification (330k), it was possible to investigate clustering of nanoparticles. The image clearly shows the separation of the nanoparticles of a cluster enveloped by a possible passivation layer.





Figure 1 shows a typical SEM microstructure of cast SmCo₂Cu₃ magnets with EDS chemical composition analysis. There are many black dots in the right grain in figure 1a. EDS results found these dots had the same composition as matrix-1:5 phase, which means that these dots are originally from matrix phase. In higher magnification SEM images, some irregular particles were observed near the grain boundary (figure 1b). From EDS chemical composition analysis (table1), particle 1, particle 5, and particle 6 are the pieces of matrix phase, whereas particle 2, particle 3, and particle 4 show very high Sm content and considerable O content, which indicates that these particles are Sm oxides.



Fig. 1a. Typical SEM microstructure of cast SmCo₂Cu₃ magnet



Fig. 1b. SEM image near grain boundary in cast SmCo₂Cu₃ magnet

Particle	Sm(at%)	Co (at%)	Cu (at%)	O (at%)
1	20.1	28.4	50.6	0.9
2	87	1.3	5.6	6.1
3	92	1.5	2.2	4.3
4	90.9	1.5	6.9	0.7
5	21.6	28.8	48	0.6
6	20.5	32.6	46.2	0.7

Table 1 EDX chemical composition analysis of different particles in SmCo₂Cu₃ cast magnet

Since the installation of the high powered SEM, various types of methods in the production of nanoparticles can now be characterized. Conventional methods, such as deposition using standard sputtering techniques and chemical deposition are being investigated to produce nanoparticles. The SEM has been used to analyzed these various techniques, and improvements in the production of nanoparticles have been achieved. Below is a SEM image of TiO2 film chemically deposited on a Si substrate.



TiO2 clusters showing particles between 10 to 50 nm

Numerous programs in other departments including Material Science, Mechanical Engineering, Chemical Engineering, Geology, and Chemistry, have benefited from the presence of a modern SEM. In addition, the microscope has offered the type of educational training in state of the art microscopy and microanalysis skill needed for our student and postdoctoral fellows. Upon completion of the installation, work proceeded in making the new Scanning Electron Microscope available for remote operation via the Internet. This has been accomplished and demonstrations were given to local High School Science teachers. Remote microscopy is an emerging technology that will make collaboration with distant colleges easier. The JEOL JSM 6335F is capable of remote operation anywhere an Ethernet connection is available.

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