ISSN (Online) 2456 -1304

International Journal of Science, Engineering and Management (IJSEM)
Vol 2, Issue 6, June 2017

Magnetization Studies of Aqueous Plasma Synthesized FeCu Micro-particles

[1] Naidu Seetala, [2] Deidre Henderson, [3] Alexander Ulrich, and [4] Chester Wilson
[1,2] Department of Mathematics and Physics, Grambling State University, Grambling, LA 71245, USA
[3,4] Institute for Micromanufacturing, Louisiana Tech University, LA 71272, USA

Abstract:- **Aqueous plasma techniques were used to synthesize FeCu micro-particles for the use as precursor particles for 3D printing to make miniature transformers. The magnetic properties of FeCu with five different compositions (1:0, 4:1, 1:1, 1:4 , and 0:1) were studied using VSM. Both magnetization and coercivity increased with Fe content as expected. The particle size and elemental composition were studies using SEM and EDX analysis, which were used to estimate magnetization values. The average particle size is around 0.5 µm obtained from SEM and composition from EDX of 100% Cu sample showed almost all Cu was oxidized and the EDX of 100% Fe showed some Fe was oxidized during aqueous plasma process. For all other compositions, the amount of oxygen found in EDX analysis was first accounted for oxidation of Cu. The balance was then used to oxidize part of Fe into the most stable compound, Fe2O3, and the rest of Fe was considered to be in pure metal form. Using the composition of Fe and Fe2O3 and their bulk saturation magnetization values, the expected magnetization were estimated for all compositions. The estimated magnetization values are much higher than the experimental values for all compositions. In accounting for the difference, 10% can be accounted for the particle size as surveyed from the literature, however the experimental results showed a difference of more than 10%. This could be as a result of the superparamagnetic nature at sub-micron/nano-size particles and the coercivity dependence on percent Cu suggests an exchange coupling between Fe and Fe2O3 magnetic phases within particles.**

***Index Terms*— Aqueous plasma synthesis, Magnetization, FeCu micro-particles**

I. INTRODUCTION

Additive manufacturing [1] or rapid prototyping is an advanced manufacturing process to build material by adding powders layer by layer and local heating using Selective Laser Melting (SLM) [2]. SLM is the forefront of new rapid prototyping for large companies. The SLM machines take metal powder from a reserve and spread them out onto a stage one layer at a time to be patterned by a high-power laser under the direction of Computer Aided Design (CAD) software. The purpose of this investigation is to find a more economical method for the generation of bi-metallic FeCu micron-size powder which will be used as 3D-printing feedstock to make magnetic material for miniature transformers. Here, we used aqueous plasma techniques to synthesize FeCu micro-particles at different compositions (Fe:Cu = 1:0, 4:1, 1:1, 1:4, and 0:1) to manipulate the magnetic properties of the product. Usually, plasma based synthesis of metal particles produce nano-scaled particles [3], however we modified the aqueous plasma synthesis techniques to produce micro-scale particles.

II. EXPERIMENTAL PROCEDURES

A. Aqueous plasma Synthesis

A 100-mL beaker was filled to 80 mL with a solution of0.25 M copper chloride and 0.25 M iron chloride. The role of the metal salts is to increase conductivity and facilitate ion transfer for the next step. Fig. 1 shows the schematic of the aqueous arc plasma synthesis setup to make bi-metallic micro-particles. One copper cathode and one iron anode were placed in the solution equidistant from a titanium anode in the solution. A current control source was used to apply 10 volts at 500 mA to each of the cathodes. The most important part of the current control is that the amperage is kept the same for both the copper and iron electrodes. A second circuit was employed in conjunction using an EMCO Q30N-5 step up transformer with an applied 5 volts to generate a 3 kV DC plasma between a platinum wire 0.25 mm in diameter and a titanium counter electrode. The platinum wire was suspended less than 5 mm above the surface of the solution so that it could form the arc discharge plasma as shown in Fig. 1.

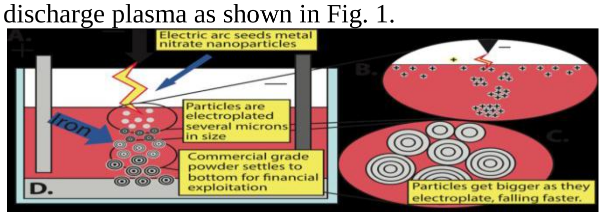

Figure 1: Schematic of aqueous arc plasma synthesis setup to make micro-particles.

ISSN (Online) 2456 -1304

International Journal of Science, Engineering and Management (IJSEM)
Vol 2, Issue 6, June 2017

The purpose of the plasma is to form nanoparticle seeds for the metal cathodes to build on top of. The solution underwent vigorous stirring while both processes were occurring. The experiment was run five separate times. The first run only applied current to the copper cathode. The second step was to only apply current to the iron cathode. These first two steps were done to show both ends of the alloy spectrum. The next three steps applied varying current ratios to the copper and iron cathodes in order to create copper-iron alloys that are 1 to 4, 1 to 1, and 4 to 1 by atomic percent.

B. Particle size and elemental analysis

The particle size distribution was studies using a FEI Quanta 200 Environmental Scanning Electron Microscope (SEM) and the elemental composition using EDAX Energy Dispersive x-ray (EDX) detector. The elemental composition was used to estimate the level of oxidation during the synthesis of particles and calculate the expected magnetization values.

C. Magnetization studies

The magnetic properties of FeCu micro-particles with five different compositions (1:0, 4:1, 1:1, 1:4, and 0:1) were studied using Model 880-CTS Digital measurement system Vibrating Sample Magnetometer (VSM). The maximum applied magnetic field was 10,000 Gauss. The composition from EDX of 100% Cu sample showed almost all Cu was oxidized and the EDX of 100% Fe showed some Fe was oxidized during aqueous plasma process. For all other bimetallic composite particles, the amount of oxygen found in EDX analysis was first accounted for oxidization of Cu. The balance oxygen was then used for partially oxidizing Fe into the most stable compound, Fe_2O_3, and the rest of Fe is considered to be in pure metal form. Using the composition of Fe and Fe_2O_3 and their bulk saturation magnetization values (218 emu/g for Fe and 78 emu/g for Fe_2O_3) [4-6], we estimated the expected magnetization for all bimetallic composite particles.

III. RESULTS AND DISCUSSION

Fig. 2 shows the SEM viewgraph of aqueous arc plasma synthesized FeCu micro-particles. The average value for particle size is around 0.5 µm. The details of the average size from SEM and the elemental composition from EDX are presented in Table 1 for five different particles synthesized with different intended Fe/Cu compositions. The elemental composition obtained from the EDX analysis for the intended 100% Cu particles showed almost all Cu was oxidized while for the intended 100% Fe particles showed that Fe is partially oxidized during aqueous arc plasma synthesis process. This observation helped us to use the EDX elemental composition for bimetallic FeCu particles to estimate the degree of Fe oxidation for each set of composite particles and there by estimate the expected magnetization as described in the

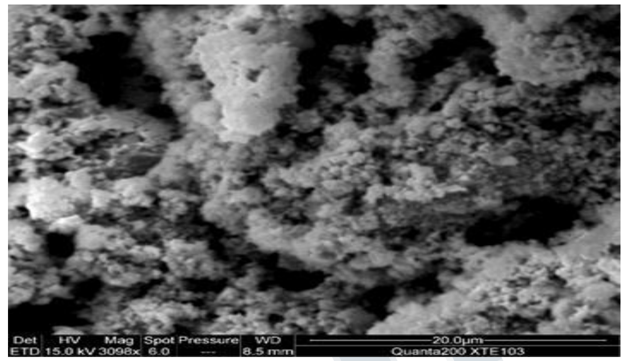

Figure 2: SEM viewgraph of FeCu micro-particles

Table 1: SEM and EDX analysis of FeCu micro-particles.

Particles Synthesized	Size (µm)	Elemental composition from the EDX analysis
100%Fe	0.42	76.76%Fe 23.24%O
80%Fe20%Cu	0.59	72.1%Fe 14.33%Cu 13.57%O
50%Fe50%Cu	0.31	51.9%Fe 32.71%Cu 15.93%O
20%Fe80%Cu	0.32	17.1%Fe 72.58%Cu 10.33%O
100%Cu	0.44	88.74%Cu 11.26%O

The magnetic hysteresis curves obtained from VSM for all FeCu micro-particles are shown in Fig. 3. Magnetization and coercivity, both, increased with Fe content as expected.

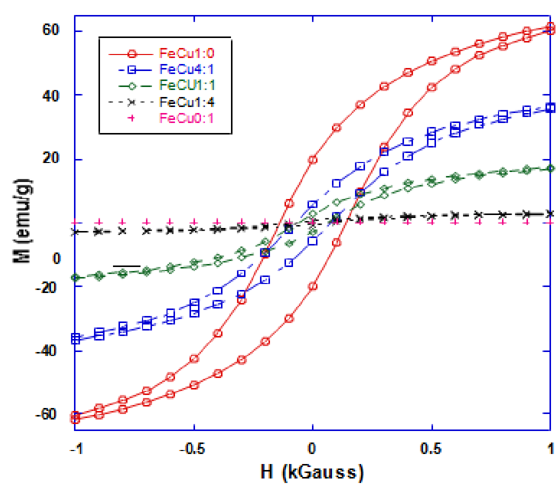

Figure 3: Magnetization M of FeCu micro-particles with applied magnetic field H.

ISSN (Online) 2456 -1304

International Journal of Science, Engineering and Management (IJSEM)
Vol 2, Issue 6, June 2017

and the expected saturation magnetization values from the estimates made from the oxidized and non-oxidized Fe amounts calculated from the EDX elemental compositions and saturation magnetization values of bulk pure-Fe and Fe2O3 [4-6].

Table 2: Magnetization results from VSM and the saturation magnetization estimates from EDS analysis using oxidation levels and bulk magnetization values.

Particles Synthesized	**M (emu/g) @10 kGaus**	**Coercivity H_c**	**M_s(emu/g) EDX estimates**
Fe	76.84	138.7	108.1
Fe80Cu20	50.60	74.1	106..0
Fe50Cu50	24.22	60.0	52.
Fe20Cu80	3.6	60.0	6.
Cu	0.006	-	0

The estimated magnetization values are much higher than the experimental values for all compositions. In accounting for the observed differences, 10% can be accounted for the particle size as surveyed from the literature [7], [8]. However, the experimental results showed more than 10% difference with much lower experimental values compared to the estimates calculated from the EDX analysis of Fe oxidation levels. This could be because of the superparamagnetic nature at sub-micron/nano-size particles, and there may be an exchange coupling between the magnetic phase of Fe layer and the magnetic phase of Fe2O3 layer within a particle. The nonlinear behavior of magnetic coercivity with the increase in Cu percentage as shown in Fig. 4 suggests the existence of the exchange coupling between two phases. The spin exchange coupling, especially between hard and soft magnetic phases, has shown advantage in developing new type of materials such as high resistivity magnets, composite magnets with low eddy current loss and improved high frequency characteristics [9].

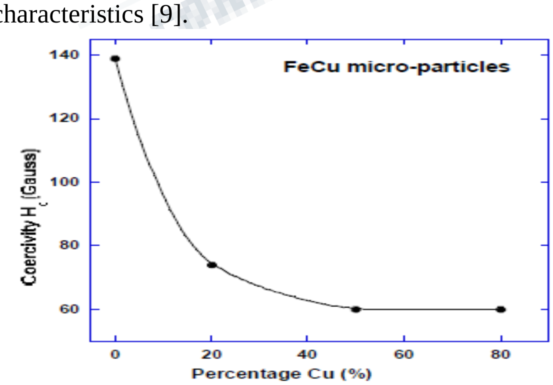

Figure 4: Hc vs %Cu in FeCu micro-particles.

IV. CONCLUSIONS

Bimetallic FeCu micron size (around 0.5 µm diameter) particles were synthesized by aqueous arc plasma techniques for the use as precursor particles for 3D printing technology to make miniature transformers. The estimated magnetization values are much higher than the experimental values for all elemental compositions observed by EDX after accounting for the oxidation level of Fe in each set of composite particles.

In accounting for the differences, 10% could be accounted for the particle size effect as surveyed from the literature, however the experimental results showed a difference of more than 10%. These differences may be assigned to the super-paramagnetic nature at sub-micron/nano-sized particles and nonlinear dependence of coercivity with percent Cu in FeCu particles suggests an exchange coupling between Fe and Fe2O3 magnetic phases within a particle.

V. ACKNOWLEDGMENTS

Work is supported by NSF-EPSCoR Consortium for Innovation in Manufacturing and Materials (CIMM) cooperative agreement OIA-15410795 and the Louisiana Board of Regents.

REFERENCES

[1] S. H. Huang, P. Liu, A. Mokasdar, and I. Hou, “Additive manufacturing and its societal impact: A literature review. International Journal of Advanced Manufacturing Technology”, Int. J. Adv. Manuf. Tech., vol. 67, pp. 1191–1203, 2013.

[2] H. Schleifenbaum, W. Meiners, K. Wissenbach, and C. Hinke, “Individualized production by means of high power selective laser melting”, Cirp J. Manuf. Sci. Tech., vol. 2, pp.161–169, 2010.

[3] A. M. Orlov, I. O. Yavtushenko, D. S. Bodnarskii, and N. V. Ufarkina, “Production of metal nanoparticles from aqueous solutions in the arc plasma”, Technical Physics, vol. 58, pp. 1267–1273, 2013.

[4] W. Gong, H. Li, Z. Zhao, and J. Chen, “Ultrafine particles of Fe, Co, and Ni ferromagnetic metals”, J. Appl. Phys., vol. 69, pp. 5119-5121, 1991.

[5] T. Klemmer, D. Hoydick, H. Okumura, B. Zhang, W.A. Soffa, Scr. Metall. Mater. 33 (1995) 1793

[6] R. Skomski, J. Phys. - Condens. Mat. 15 (2003) R841.
[7] H. M. Lu, W. T. Zheng, and Q. Jiang, "Saturation magnetization of ferromagnetic and ferromagnetic nanocrystals at room temperature", J. Phys. D: Appl. Phys., vol. 40, pp. 320–325, 2007.

[8] C. Caizer, "Saturation magnetization of γ-Fe2O3 nanoparticles dispersed in a silica matrix", Physica B, vol. 327, pp.27–33, 2003.

[9] N. Imaoka, E. Kakimoto, K. Takagi, K. Ozaki, M. Tada, T. Nakagawa, and M. Abe, "Exchange coupling between soft magnetic ferrite and hard ferromagnetic Sm2Fe17N3 in ferrite/Sm2Fe17N3 composites", AIP Advances vol. 6, 056022, pp. 1-5, 2016.