Development of stable $Fe_{16}N_2$ magnetic nanoparticles for permanent magnet applications

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The global market for permanent magnets will exceed $31B by 2020. China dominates the supply chain, and extracts and produces about 95% of the world's rare earth minerals. Since 2010, China has cut exports, creating risk in the supply chain. In addition to supply chain concerns, the extraction process is highly polluting to the environment.

Over the last 3 years, Nanofoundry, LLC has undertaken an intensive program to develop a new, non-rare earth permanent magnet. Nanofoundry's approach is an adaptation of a bench-top chemical method developed at VCU. This bench-top method generated a promising cobalt carbide (Co3C) nanoparticle material with an energy product of 17 MGOe. The particle size was less than 20 nm, and the material was a pure Co3C hard magnetic phase with a homogeneous morphological structure.

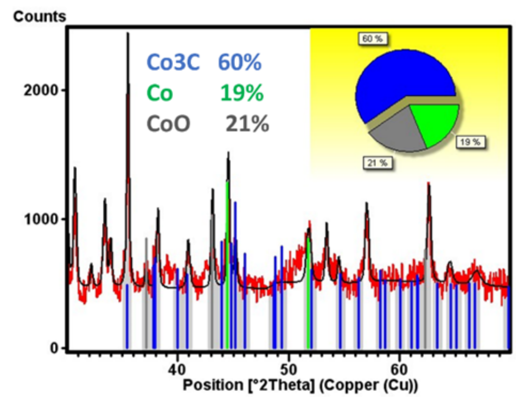

Fig. 1 XRD pattern for the particles

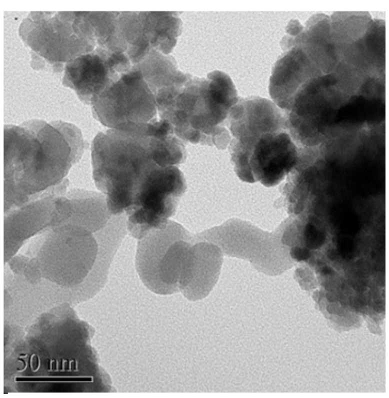

Fig. 2 TEM for the particles

Nanofoundry piloted the production of this material on its proprietary microfluidic reactor. Nanofoundry's process obtained about 60% of the carbide phase (Fig. 1) mixed with cobalt and cobalt oxide nanoparticles. The particle size was around 20 nm (Fig. 2), similar to the result obtained for the bench-top reaction. However, Nanofoundry was unable to duplicate the magnetic properties achieved in the bench-top method. Nanofoundry has tested a meaningful range of promising metal precursors, surfactants, and process configurations using a Design of Experiments framework, but has not yet been successful in producing pure Co3C particles.

We have concluded that the catalytic reaction for producing the carbide was not fully achieved in the continuous fluidic reactor. We believe that a large portion of the particles is oxidized during the reaction within the open system of the fluidic reaction. Further, preliminary trials with the iron carbide system reveal the same challenges with oxidation and incomplete reaction, which is consistent with expectations.

For the sake of completeness, we will perform one other trial based on a CoFeC system under supercritical conditions with ethanol. If successful, this carbide would present commercially useful magnetic properties (Figures 3-5; El-Gendy et al., Appl. Phys. Lett. 106, 213109, 2015) at a competitive price point. In particular, CoFeC particles have a size of less than 20 nm with a magnetocrystalline anisotropy of about 5 MJm^{-3}. This is high compared to NdFeB magnets (4 MJm^{-3}, J. App. Phys. 61(8), 3436, 1987). Additionally, CoFeC nanoparticles are thermally stable up to 790 K, which is superior to any other magnetic materials on the market.

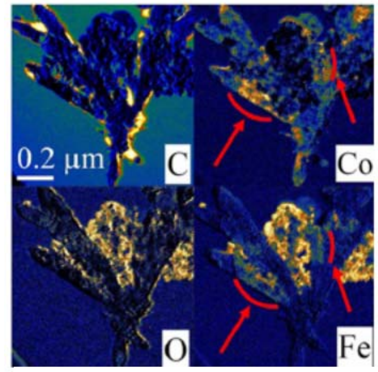

Fig. 3 TEM for the CoFeC

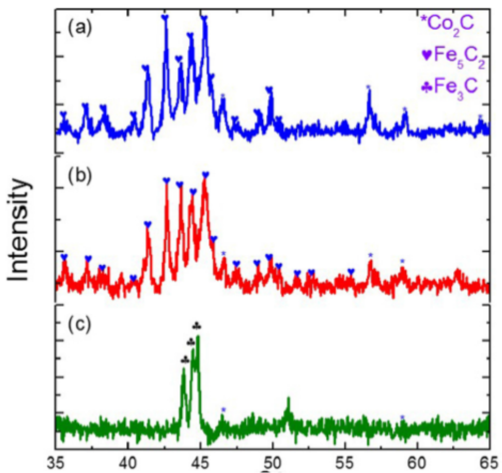

Fig. 4 XRD pattern for CoFeC

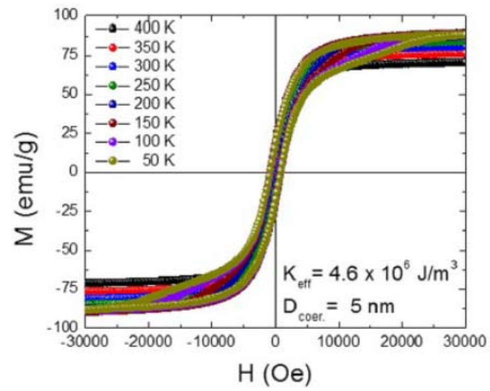

Fig. 5 Magnetic Properties of CoFeC

While these properties are very compelling, we have low confidence that the carbide system will be feasible using a continuous fluidic reactor, for the same reasons of oxidization and incomplete reaction as we have found in the CoC and FeC reactions.

However, the excellent performance of the iron, cobalt, and iron-cobalt systems does encourage us that the production of related systems may be feasible. In particular, we propose to move forward with the production of nitrides in lieu of carbides, in particular the iron nitride (Fe16N2). Potential benefits of iron nitride permanent magnets are summarized as follows:

- The theoretical magnetic energy product for the iron nitride (Fe16N2) magnet is 130 MGOe, which is more than twice the maximum reported magnet energy product of the best commercially available magnet, the rare-earth neodymium magnet.
- It is free of rare earths.
- It is low cost due to low cost inputs (iron and nitrogen), and a low-cost production process.

- It is environmentally friendly due to both the benign nature of the inputs and the inherent safety of the production process.
- Potentially high thermal stability.

We propose to validate Fe16N2 in a batch/benchtop mode, and then in a continuous-flow microreactor. We are targeting a magnetization (MS) of 200 emu/g and a coercivity (HC) of 3 kOe, which is comparable to many common commercially-available magnets.

Our proposed technical approach for the continuous-flow process (Fig. 6) is as follows: We will use our proprietary microfluidic wet chemical synthesis process to produce oxide input materials, followed by a post-annealing nitridation process. We will use ball milling and supercritical conditioning to tune size distribution and magnetic properties.

The rationale for this process is that the nitridation process eliminates the problems with oxidation and incomplete reaction found with the carbides. Ball milling produces nanoparticle material, which drives up the magnetic anisotropy (1x 10^6 MJm^{-3}). Because Fe and N are abundant, the materials cost is low and the process is otherwise cost-effective and sustainable.

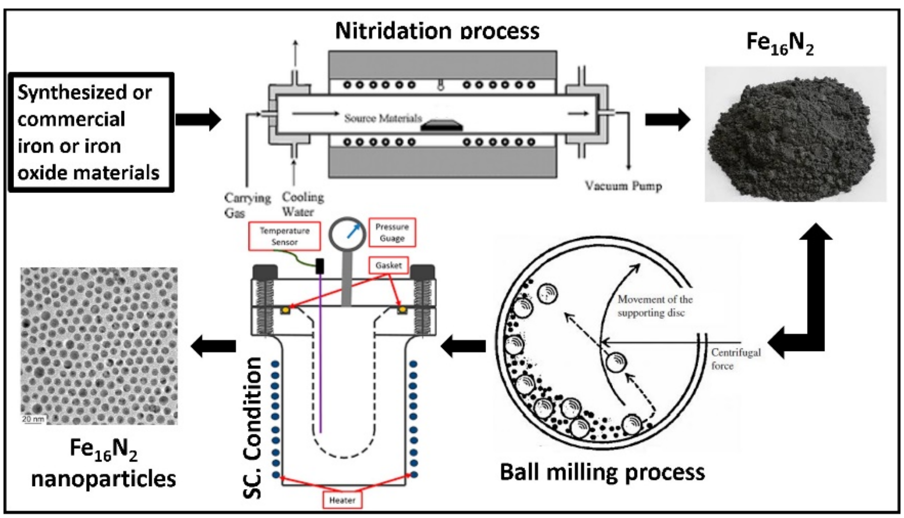

Fig. 6 Schematic diagram for the production process of $Fe_{16}N_2$ nanoparticles

Nanofoundry, LLC has deep expertise in chemistry, microfluidic process synthesis, and magnet science. By combining our experience and with our microfluidic reactor capabilities, we are uniquely positioned to develop a new non-rare earth magnet material which environmentally friendly and has an excellent performance to price ratio. Beyond the advancement of the materials technology itself, a key outcome of this work will be the development of a new process technology and the characterization of new magnet materials.