Phase sensitive synthesis of iron oxide nanoparticles by microwave plasma torch

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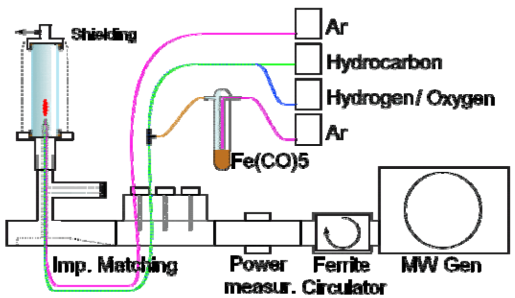
Introduction

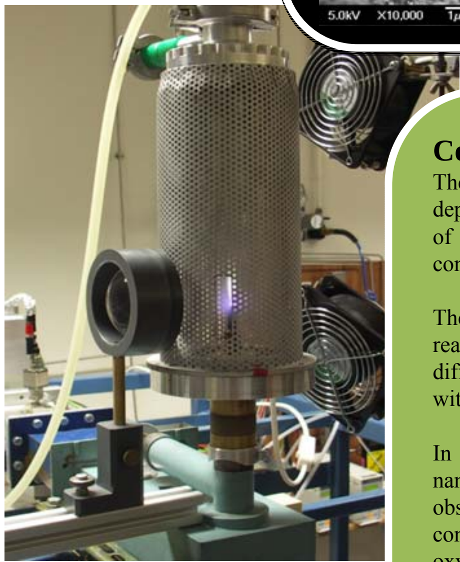
- During past few years there has been an increasing interest in the controlled synthesis of iron oxide nanoparticles (NPs) because of broad range of their applications.
- We synthesize iron oxide NPs in microwave plasma torch at atmospheric pressure - it could be cheap, reliable and ecological alternative to conventional chemical methods.
- Focuse on the production of maghemite ($\gamma - Fe_2O_3$) NPs - ferrimagnetic properties for the diameter above 10–20 nm and superparamagnetic behavior below; good biocompatibility and chemical stability $\Rightarrow$ applications in ferrofluids, catalysis, high-density magnetic recording, drug delivery and magnetic resonance imaging (MRI) as contrast agents.

Microwave Torch Experiments

Synthesis of iron based nanoparticles in microwave plasma axial torch:

- mw power of 180W,
- atmospheric pressure,
- mixture of Ar (670 sccm), variable flow rate of $Fe(CO)_5$ vapors, optional addition of O_2 or H_2.





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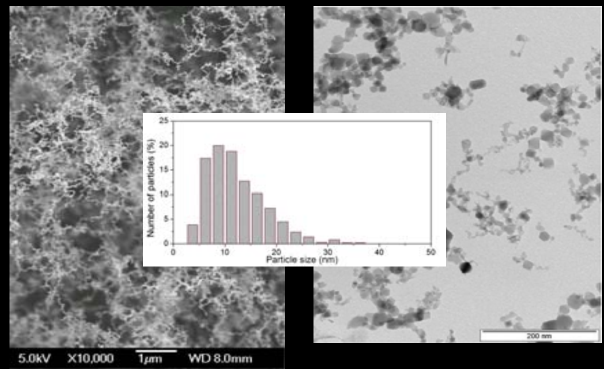
SEM and images with particle size distribution.



Conclusion

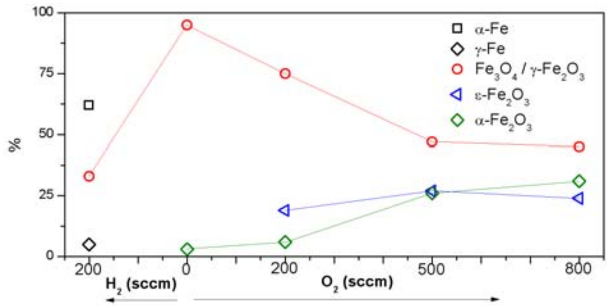
The optimization of the torch reactor design and deposition conditions allowed continual synthesis of maghemite nanoparticles at low power consumption with small fraction of other phases.

The synthesized powder was collected at the reactor walls and analyzed by TEM, X-ray diffraction, Raman and infrared spectroscopies without any further purification or treatment.

In pure argon, the powder consisted of $\gamma - Fe_2O_3$ nanoparticles. The mean diameter of NPs, as observed by TEM, was 12 nm with a 90% confidence interval 5.5–22 nm. The addition of oxygen gas resulted in multiphase composition of synthesized powder ($\gamma - Fe_2O_3$, $e - Fe_2O_3$ and $a - Fe_2O_3$). The amount of latter two phases increased with increasing oxygen flow rate.

Experiments and Results

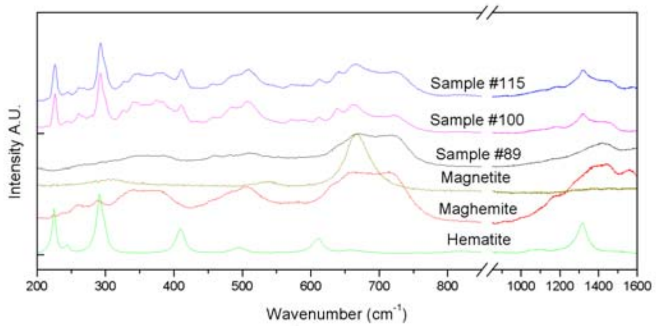
1. Influence of H_2 or O_2 addition on the phase of iron and iron oxide nanoparticles (NPs) for $Fe(CO)_5$ flow rate of 0.05 sccm.
2. Optimum conditions for sythesis of pure maghemite NPs found for $Fe(CO)_5$/Ar plasma (no addition of H_2 or O_2).
3. Influence of increasing precursor flow rate on the phase composition in case of the previously found optimal conditions.



XRD spectra evaluation showing dependence of phase composition on added hydrogen or oxygen into reaction chamber. It's clear that optimal conditions for maghemite synthesis are in pure precursor/argon mixture. XRD cannot distinguish between maghemite or magnetite.

Pure $Fe(CO)_5$ in argon synthesis and products by XRD

Sample	α-Fe_2O_3		γ-Fe_2O_3/Fe_3O_4		Iron pentacarbonyl gas flow
	Amount	Mean size	Amount	Mean size	
#89	-	-	100%	20nm	0.05 sccm
#100	3%	55nm	97%	25nm	0.2 sccm
#115	10%	80nm	90%	40nm	1 sccm



With increasing flow rate production we also increase production of hematite as seen from XRD analysis. We have also used Raman spectroscopy to determine maghemite/magnetite from which is clear that smaple is maghemite based with hematit which was already seen from XRD.