

PROCESSING OF METALLIC FILTERS BY POWDER METALLURGY TECHNIQUE

W. Daoush, S. Moustafa, S. Kayetbay

Abstract

In this work nickel coated iron oxide (Fe_2O_3) pellets of different particle sizes ranging between 500~900 μm were prepared using chemical reduction method. Hypophosphite bath in alkaline tartarate was used as a chemical coating bath for Fe_2O_3 particles with Ni, then sintered at 1000 °C in a reducing atmosphere for 60 min to produce metallic filters having a composition of Fe70 wt.-%-Ni 30 wt.-% in the shape of cylindrical candles.

Extensive metallographic studies using optical and scanning electron microscopy were carried out. It was found that the coating layer of the nickel on the iron oxide powder is about 250 μm thick. Sintering of the coated powder takes place by neck growth formation between the coated pellets. The magnetic properties of the iron oxide pellets, the Ni coated ones and the related sintered Fe-Ni filters were measured by a vibrating sample magnetometer (VSM). The results indicate that the reduced Fe_2O_3 to Fe has a saturation induction (B_s) value of 1.47 (electromagnetic unit/gram) emu/g which form a solid solution Fe-Ni with the Ni layer having a B_s value 129.6 emu/g and coercive force (H_c) value of 16.5 (Orested) Oe. The porosity values, as determined by the Poresizer apparatus, were between 27.5% and 41.5% corresponding to 5.5 g/cm³ and 3.1 g/cm³ density respectively.

Keywords: *porous materials, filters, sintering and magnetic properties*

INTRODUCTION

Metal filters are available in a wide range of materials including copper, nickel, bronze, stainless steel and "Monel", and are widely used for the filtration of fuel oils, chemical solutions and emulsions. They are also efficient in separating liquids of varying surface tension. The unreliability of the ceramic filter elements in demonstration trials and the high capital cost of these systems have hindered their application and are factors restricting the uptake of gasification power plants in general. A successful development of a durable metallic filter system for the gasification and purification would be a major step towards its implementation. Metallic filter elements have potential applications in all systems and in other industries requiring hot gas cleaning.

The development and introduction of hot gas cleaning (HGC) technologies offer the potential of a lower cost approach to pollutant control, leading to simpler cycle configurations with associated efficiency advantages. Hot gas filtration has not only been adopted as an essential system component in technologies, but it is also being used to remove particulates prior to water scrubbing of fuel gases in first generation plants. The filters currently employed are based on the designs developed for pressurized fluidized bed combustion applications in the 1980s using ceramic filter elements. The unreliability of the

ceramic filter elements in demonstration trials and the high cost of these systems have hindered their application and are factors restricting the uptake of gasification power plants in general, [1-5].

Metallic filter media provides a number of significant advantages over ceramics such as, Lower pressure drop - leading to a reduced filtration area and hence reduced capital cost, excellent cleanability, more predictable durability and reliability and simpler installation and handling requirements

The potential for fuel gases to cause sulphidation, erosion and fouling raises concerns over the selection of materials and the lifetimes of filter components, similar to those for the heat exchanger which is used to cool the fuel gas before the hot gas cleaning stages. Also down-time corrosion, resulting from deposits of particles and condensates which develop during operation, may lead to severe pitting damage and stress corrosion cracking. Micro-filter is a world class manufacturer of metallic filter media for a wide range of applications, [6,7].

Various applications for metallic filters are covered, including petrochemical, agricultural fluid and gas transfer. Many of these are designed for safety applications such as those required in hydraulic lines typically fluid control and cleaning.

The material of filters covered Iron, Nickel and Cobalt based alloys and included a number of welded specimens. Baseline alloys were also included to provide an indication of the relative corrosive nature of the environment. The list of materials included in the screening test along with a nominal chemical composition. Materials were tested in forms relevant for filter manufacture such as with a weld or pre-treatment such as pre-oxidation.

The objective of this study is to design and develop metallic filters having uniform, closely controlled porosity using a narrow range particle size of bimetal powders, namely Fe coated with Ni layer.

EXPERIMENTAL

The iron oxide (Fe_2O_3) palletized powders of 0.9, 0.7 and 0.5 mm particle size were selected from a waste powder produced during iron wires manufacturing industries, and were chemically coated with electroless Ni in alkaline tartarate bath as listed in Table 1. The PH of the bath should be near 9 and the optimum temperature near 98°C . In the beginning of the reaction 20 mg iron oxide (Fe_2O_3) pellets were added to one liter of the Ni bath and then the hypophosphite was added step by step to the mixer with mechanical stirring of 500 rpm and the reaction was completed in about 120 min then finally the particles were washed, filtered and dried in 110°C . Nikon optical as well as (JEOL, JSM-T20) scanning electron microscopy were used for investigating the prepared powder morphology. XRD for the powders was performed using BRUKER D8 ADVANCE at room temperature.

Tab.1. The chemical composition for the electroless Ni bath.

$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	30 mg/l
Pot. Sod. Tartarate	30 mg/l
NH_4Cl	25 mg/l
Sodium hypophosphite	30 mg/l
PH	~ 9
Temperature	98°C

The prepared Ni coated iron oxide (Fe_2O_3) pellets were sintered by packing the Ni coated pellets in a fused alumina crucible of the dimension 1.5 cm diameter and 1.2 cm length in a tube furnace at 1000°C in a hydrogen atmosphere.

The magnetic properties of the iron oxide powder and the related coated one as well as the sintered filters were measured using a vibrating sample magnetometer (VSM) model 9600-1 LDJ. The measured properties included coercive force and magnetization saturation. Because the magnetization saturation and remanance changes with weight and volume of the sample, the results were divided on the sample's weight.

RESULTS AND DISCUSSION

Pellets of Fe_2O_3 powders with 0.9, 0.7 and 0.5 mm particle sizes are used as a source material for iron which was coated by electroless Ni and packed in a fused alumina crucible and sintered at 1000°C to form Fe-Ni phase with 30 wt.% Ni content.

Optical micrograph cross sectional area of electroless coated powder contains 30 wt.% Ni and 70 wt.% iron as illustrated in Fig.1a, we can show the coating layer of Ni on the spherical Fe_2O_3 powder has a 250 μm thickness but from the SEM micrograph which was illustrated in Fig.1b we can show a uniform Ni layer structure deposited on the spherical Fe_2O_3 powder.

After sintering at 1000°C in a H_2 atmosphere the Fe_2O_3 powder was reduced to Fe according the following equation: $\text{Fe}_2\text{O}_3 + \text{H}_2 \xrightarrow{1000^\circ\text{C}} 2\text{Fe} + 3\text{H}_2\text{O}$

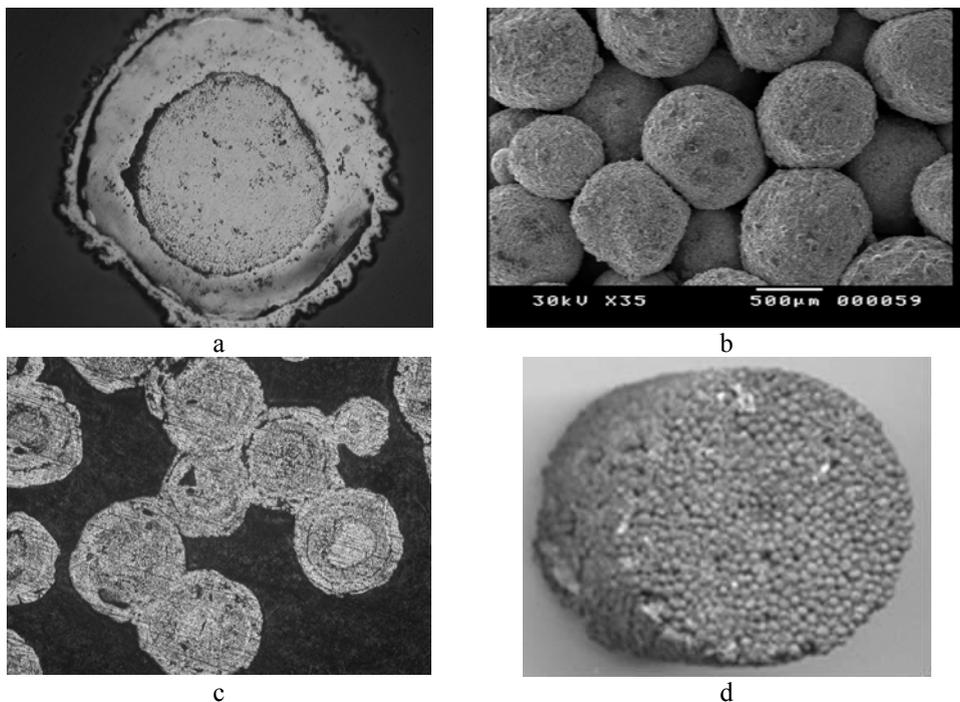


Fig.1. The microscopic graphs of (a) the prepared metallic filters where a) optical micrograph for the coated iron oxide pellets by Ni metal b) SEM micrograph for the prepared sintered Ni c) an optical micrograph for the cross section area of the prepared sintered Ni filter but d) which is a stereographic for the prepared cylindrical candle.

It is observed from Fig. 2a, the XRD pattern for the Fe_2O_3 powder itself in comparison with the XRD pattern in Fig.2b for the sintered Ni-coated powder, where FeNi is formed at 1000°C in a reducing atmosphere of hydrogen gas.

One can show from Fig. 1c an optical micrograph for the cross sectional area of the sintered FeNi , in which it was observed that the disappearance of the Ni layer was diffused in the Fe bulk to form a FeNi phase at 1000°C as illustrated in the XRD pattern in Fig.2b. We can show from Fig. 1d a stereographic picture for the final candle product with a 13 mm diameter and 10 mm length.

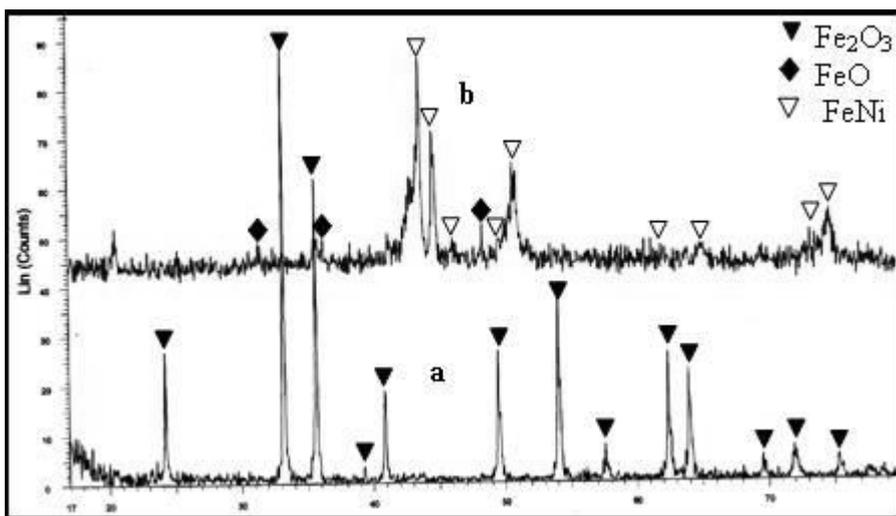


Fig.2. XRD pattern for the supported iron oxide powder and the related sintered Fe-Ni one, where a; the XRD pattern for the Fe_2O_3 powder without electroless Ni-coating but b; the XRD pattern for the FeNi which is formed after sintering at 900°C .

We can identify the sintering process and formation of the FeNi phase by measuring the magnetic properties of the iron oxide powder, the Ni-coated one and the sintered one as shown in Fig.3. The saturation induction (B_s) value of the iron oxide as listed in Table 2 is 1.47 emu/g with a coercive force (H_c) 268.9 Oe, but after coating of the iron oxide with electroless Ni the B_s value was increased to 42.34 emu/g as shown in the hysteresis curve in Fig.3, curve a, which is near to the theoretical one for pure Ni 54.8 emu/g due to the formation of the Ni layer on the iron oxide powder and the H_c is decreased to 110.7 Oe due to an increasing in the particle size of the powder by Ni layer formation. But after sintering at 1000°C in an H_2 atmosphere the B_s value increased to 129.6 emu/g as illustrated in Fig.3, curve b, which is due to the formation of the FeNi phase which is a soft magnetic material where H_c is 16.51 Oe as listed in Table 2.

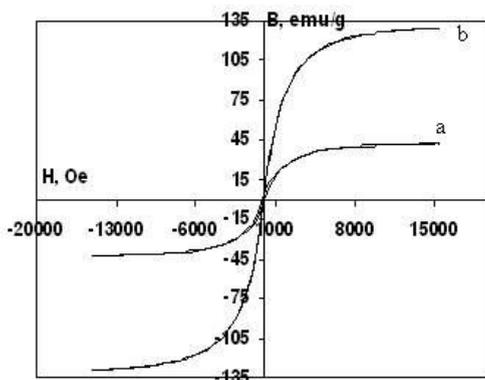


Fig.3. The B-H curve for the magnetic properties of a) electroless Ni coated Fe₂O₃ powder, and b) after reduction in hydrogen at 1000°C to form Fe-Ni (FCC).

Tab.2. The magnetic properties for the prepared coated powders and the related sintered porous materials.

Sample	B _s , emu/g	H _c , Oe
Iron oxide powder	1.47	268.90
Ni-coated powder	42.34	110.7
Reduced FeNi material	129.60	16.51

From the pore size diameter measurements for each particle size of the powder as illustrated in Fig.4 the incremental intrusion is increased by increasing the particle size of the powder which produces the sintered one. From Fig. 4 we can show two kinds of pore the first one is the pore which formed in between the channeled spherical sintered powder with a mean particle size of 50, 40 and 15 μm for 0.09, 0.07, and 0.05 mm particle size respectively, and the second kind of pores having around a 400 nm pore size on the surface of the spherical sintered powder.

One can show from Table 3 the effect of the particle size of the sintered Ni coated pellets on the sound properties, the apparent and the bulk densities were increased by increasing the particle size of the coated pellets but the porosity was decreased.

Tab.3. The sound properties for each prepared particle size for the sintered Ni coated pellets.

Sample	Bulk Density [g/cm ³]	Apparent Density [g/cm ³]	Porosity [%]
500 μm	3.1	4.2	27.5
700 μm	3.3	5.2	37.2
900 μm	5.4	9.4	41.5

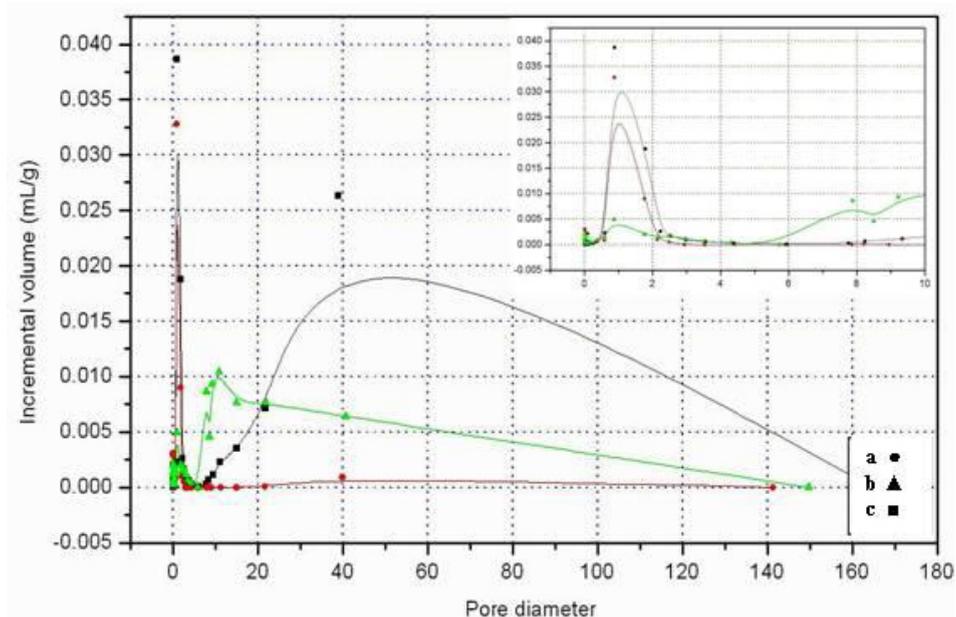


Fig.4. The incremental intrusion for mercury against the pore size diameter for the prepared filters where a) sintered powder spheres of the particle size 500 μm , b) sintered powder spheres of the particle size 700 μm and c) sintered powder spheres of the particle size 900 μm .

CONCLUSIONS

- Metallic filters with about 40% porosity in a candle shape were prepared by classical powder metallurgy technique.
- Electroless method of coating is a good technique for the preparation of metallic filters.
- The magnetic measurements can be used to identify the phase transformations at different sintering conditions
- The porosity of the metallic filters was increased by increasing the powder particle size.

REFERENCES

- [1] Takematsu, T., Maude, CW.: Coal Gasification for IGCC Power Generation. IEACR/37, IEA Coal Research, London, UK, 1991
- [2] Proc. Corrosion in Advanced Power Plants. Special Issue of Materials at High Temperatures, 14, 1997
- [3] Proc. First International Workshop on Materials for Coal Gasification Power Plant, Petten. The Netherlands, June, 1993, in Materials for Coal Gasification Power Plant, Special Issue of Materials at High Temperature, 11, 1993
- [4] Schlachter, W., Gessinger, GH. In: High Temperature Materials for Power Engineering 1990. Ed. E Bachelet et al. Kluwer, 1990, p. 1
- [5] Oakey, JE., Simms, NJ. In: Materials for Advanced Power Plant 1998. Ed. J Lecomte-Beckers et al. Forschungszentrum Julich GmbH, Germany, 1998, p. 651
- [6] Mendez-Vigo, I., Chamberlain, J., Pisa, J.: Materials at High Temperatures, vol. 14, 1997, spec.iss., p. 15
- [7] Erosion/Corrosion of Advanced Materials for Coal-Fired Combined Cycle Power Generation. Final and Summary Reports on JOUF-0022, 1994