Magnetic Properties of $SmCo_5$/Fe Exchange Coupled Nanocomposite Nanoparticles obtained by Matrix Milling and Annealing

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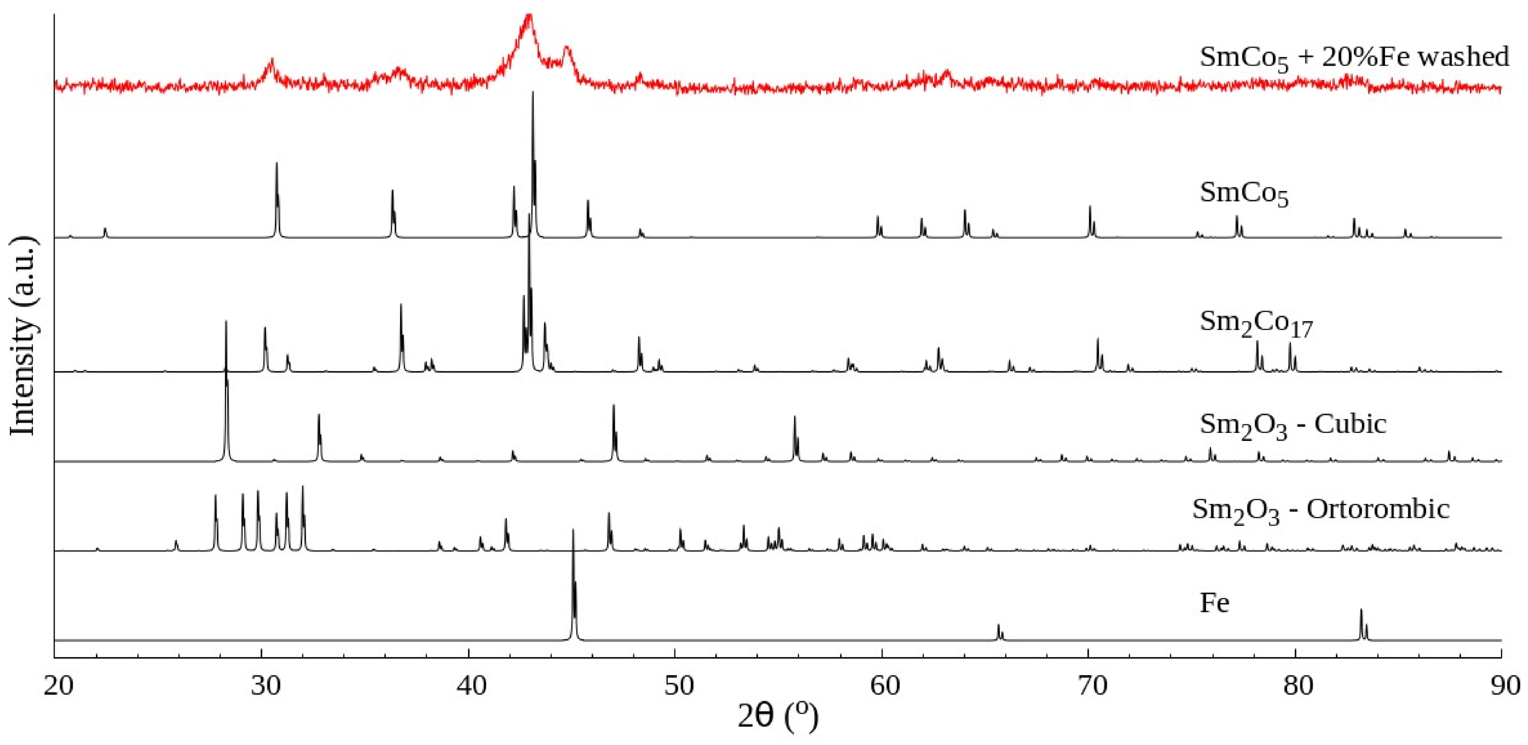
Abstract: In this work, we have produced $SmCo_5$+Fe exchange-coupled nanocomposite nanoparticles through matrix milling. $SmCo_5$ and Fe powders were first milled for six hours in a planetary ball mill; the resulting composite powders were mixed in a 60:1 weight ratio with CaO powder. This mixture was then milled for a further six hours. The nanocomposite powder was then annealed while still in the CaO matrix. After annealing, the matrix was chemically dissolved, leaving behind only the nanocomposite powder. Once separated from the matrix, the structure of the nanocomposite was studied by X-Ray diffraction, the magnetic properties of the materials were studied by measuring demagnetization curves up to 10 T and the particles morphology and chemical homogeneity were studied by scanning electron microscopy and X-ray microanalysis (EDX).

Experimental details:

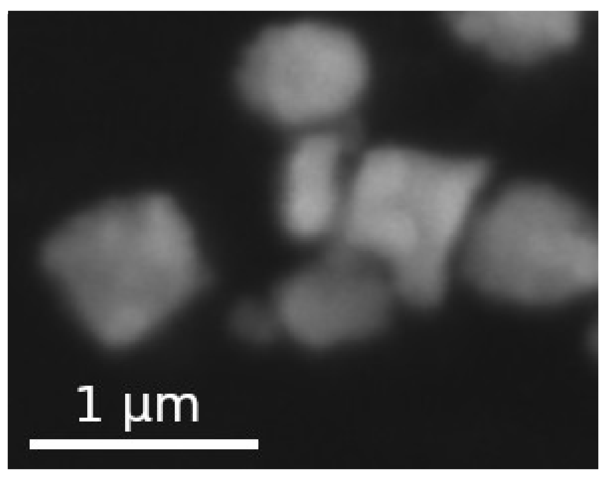
- **SmCo5+20%Fe nanocomposites** were produced by co-milling the aforementioned powders. The powders were weighed and mixed for 30 minutes in a Turbula mixer. The mixed powders were placed in a 80 ml 440C steel vial, together with 26 steel balls, 10 mm in diameter. The powders were milled for six hours in a Fritsch P4 planetary ball mill. The vial-to-main disk speed ratio was -900/333.
- After the formation of the nanocomposite, some of the powder was mixed with **CaO** in a 1:60 weight ratio, and milled for an additional two hours.
- The milled mixture was then **annealed** in high vacuum **at 600 °C for 0.5 hours**.
- After annealing, the nanocomposite particles were **separated from the CaO matrix by successive washing** with a 50:50 solution of ethanol and glycerine, in order to dissolve the CaO matrix. To favour separation, the mixture was agitated by hand and in an ultrasonic bath. The powders were washed five times for 30 minutes.
- **X-Ray diffraction** measurements were performed on a Bruker D8 Advance diffractometer equipped with a Cu source,
- **Magnetic measurements** were carried out on powder fixed in epoxy resin at 4 K in fields up to 10 T.
- **All powder handling was done under purified Ar atmosphere.**

Results and Discussions:



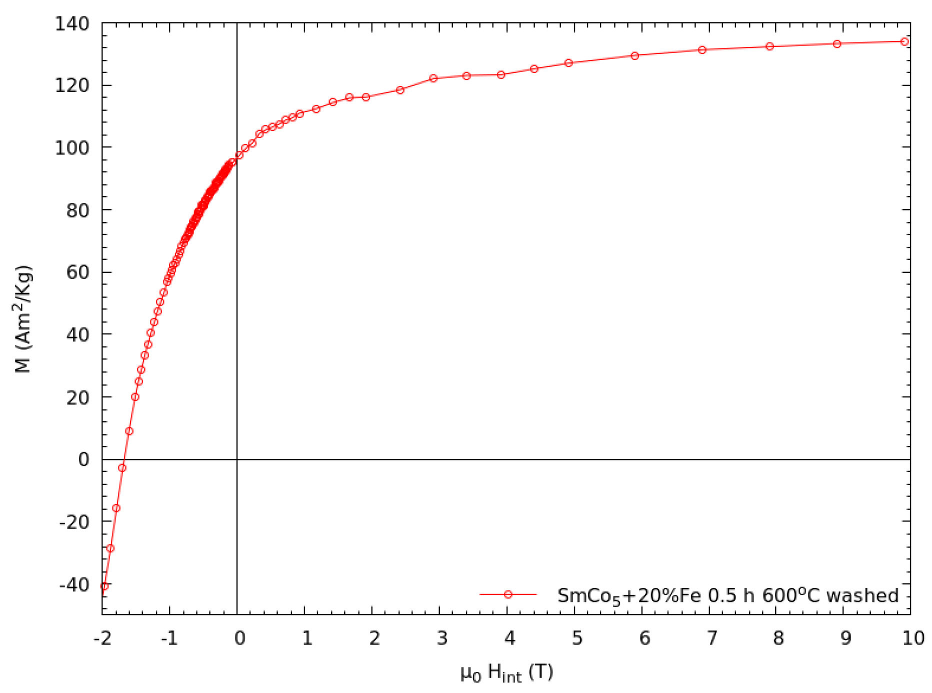
X-Ray diffraction pattern for $SmCo_5$+20%Fe nanocomposites produced by mechanical milling and CaO matrix annealing at 600 °C for 30 minutes.

XRD patterns show that the produced powders are a nanocrystalline material containg $SmCo_5$ and Fe as primary phase. Calculated XRD patterns for SmCo5 and Fe are given for reference along with Sm oxides and Sm_2Co_{17}. Due to the low signal to noise ratio, the presence of the Sm_2Co_{17} can not be adequately confirmed or excluded.
On the other hand, in spite of the low signal to noise ration we can conclude that the produced powders are not significantly oxidized during the washing process.

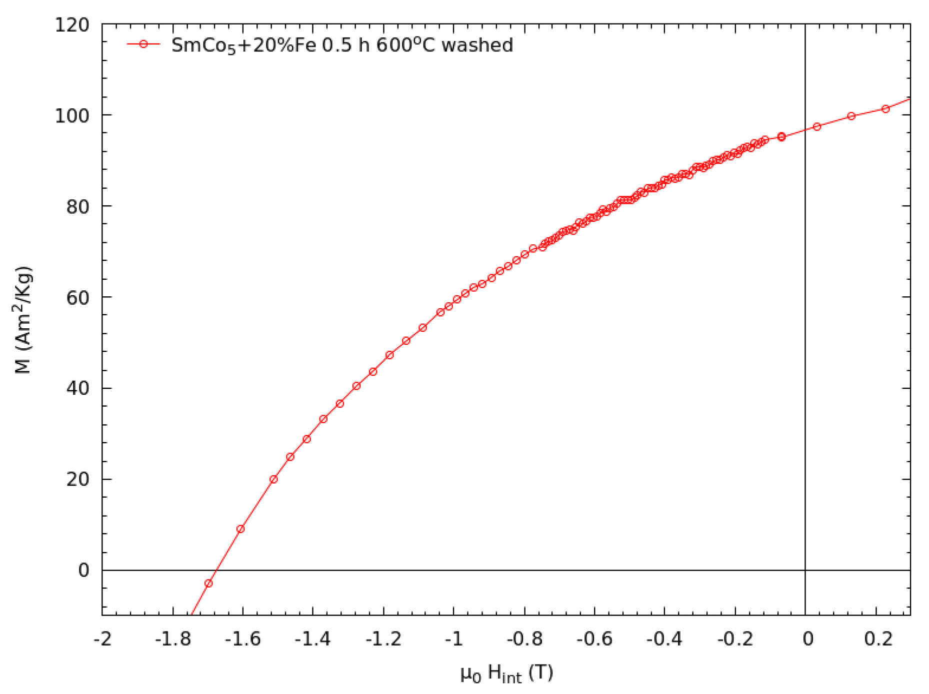


Scanning electron microscope image of $SmCo_5$+20%Fe nanocomposite particles produced by mechanical milling and CaO matrix annealing at 600 °C for 30 minutes.

SEM images show that the produced particles have diameters of under one micron. Recorded EDX spectra showed that the particles are made up of Sm, Co and Fe. In the investigated areas, the presence of CaO could not be confirmed.



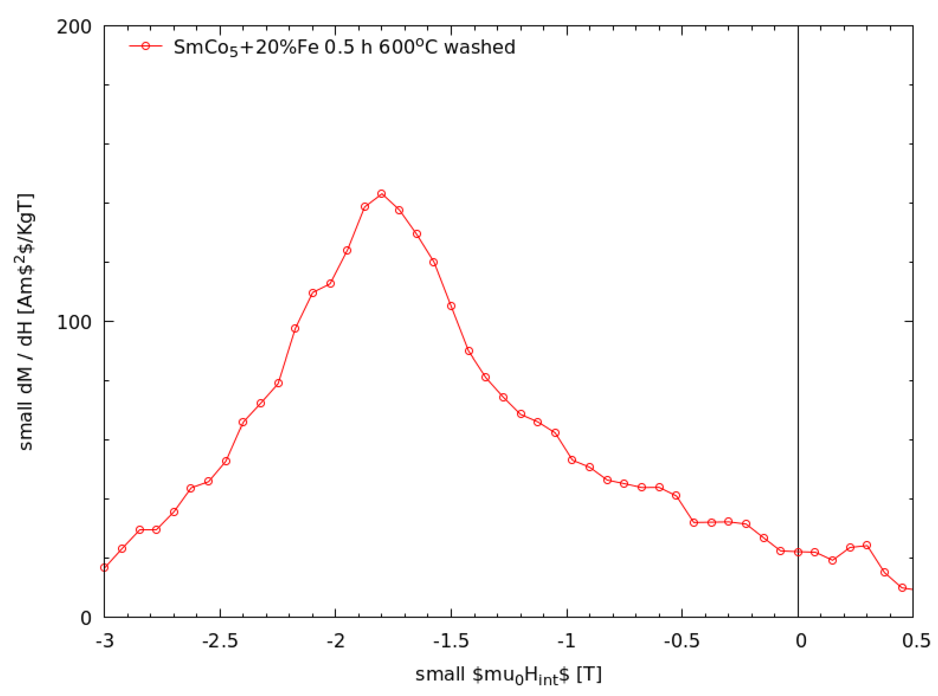
Demagnetization curve recorded at 4 K for $SmCo_5$+20%Fe nanocomposites produced by mechanical milling and CaO matrix annealing at 600 °C for 30 minutes.



Zoomed demagnetization curve recorded at 4 K for $SmCo_5$+20%Fe nanocomposites produced by mechanical milling and CaO matrix annealing at 600 °C for 30 minutes.

The magnetic measurement was carried out at 4 K in order to better evidence the degree of interphase exchange coupling. This is due to the fact that for exchange coupled nanocomposites the size of the soft magnetic phase can not exceed twice the size of the magnetic domain wall width of the hard magnetic phase. As the size of the domain wall is inversely proportional to the magnitude of the anisotropy parameter, the adequacy of the interphase exchange should be most evident at low temperature, where the domain wall width is smallest (approximately 3 nm for $SmCo_5$).

From the demagnetization curve we can see that there are no kinks around 0 T and the curve itself is very smooth, which is indicative of a good degree of interphase exchange. Moreover, the sample presents a large coercive field of 1.7 T, significantly higher than that obtained for samples annealed without the addition of the CaO matrix (1.5 T) [1], although the remanence is comparable. The ratio Mr/Ms was also evaluated for the washed particles and was found to be 0.7, which also points to a good degree of interphase exchange,
The energy product of the fully dense material was estimated to be 142 kJ/m³.



dM/dH vs H plot at 4 K for $SmCo_5$+20%Fe nanocomposites produced by mechanical milling and CaO matrix annealing at 600 °C for 30 minutes.

The interphase exchange coupling was also investigated by dM/dH plots. Peaks at high field are indicative of a good degree of interphase exchange. From the dM/dH plot, we can see that there is no peak centered around 0 T, with only one intense peak centered around 1.7 T being visible. This behavior shows that within our sample we have a good degree of interphase exchange.

Conclusions:

- Matrix milling and annealing is a viable method for obtaining sub micron particles of exchange coupled nanocomposites.
- XRD and magnetic measurements show that the phase composition and magnetic properties of the exchange-coupled nanocomposites are performant even after the washing process, as they have a high coercive field of 1.7 T and a promising energy product of 142 kJ/m³.
- It may be possible that by increasing milling times, the particle size could be reduced.

References

[1] R. Hirian, A. Bolinger, O. Isnard & V. Pop (2018), Powder Metallurgy, DOI: 10.1080/00325899.2018.1531582

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