Magnetic properties of exchange-coupled nanocomposites obtained by milling α-Fe and recycled $SmCo_5$

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Background and Introduction:

Exchange spring magnets {SPM} consist of hard and soft magnetic materials in a diffuse nano-scale composite. In SPM, exchange-coupling between neighbouring soft/hard crystallites can improve energy product {$(BH)_{max}$} when the structure, microstructure, and geometry meet certain criteria [1]. We aim to create functional nanocomposite SPM that use recycled materials. Use of recycled materials can minimize the footprint and environmental costs of the critical precursors; this is in addition to savings accrued by the technical advantages of SPM alone – all such improvement constitutes the plinth of our work towards a green-energy applications. [2]

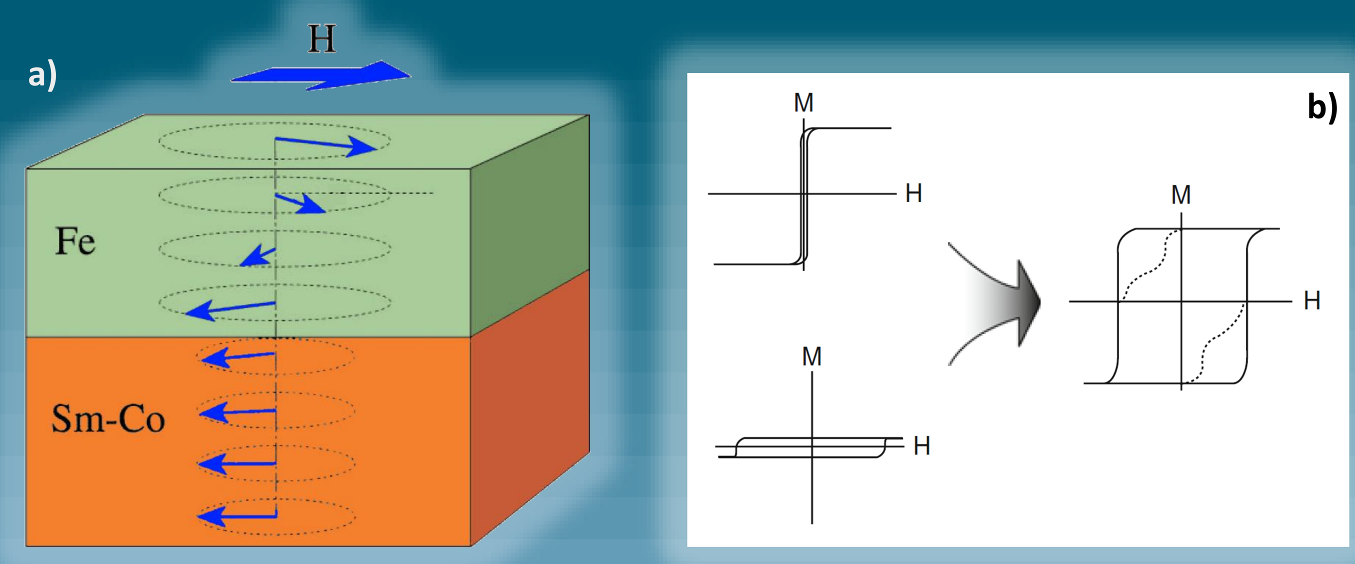

Fig.1 (a) Direction and magnitude of local moment under an external field is influenced by the critical lengths required in the structure of SPMs. [3] (b) The overall effect of exchange coupling at this length scale is to then enhance remanence without a complete sacrifice of coercivity. The dotted line represents decoupled behaviour. [4]

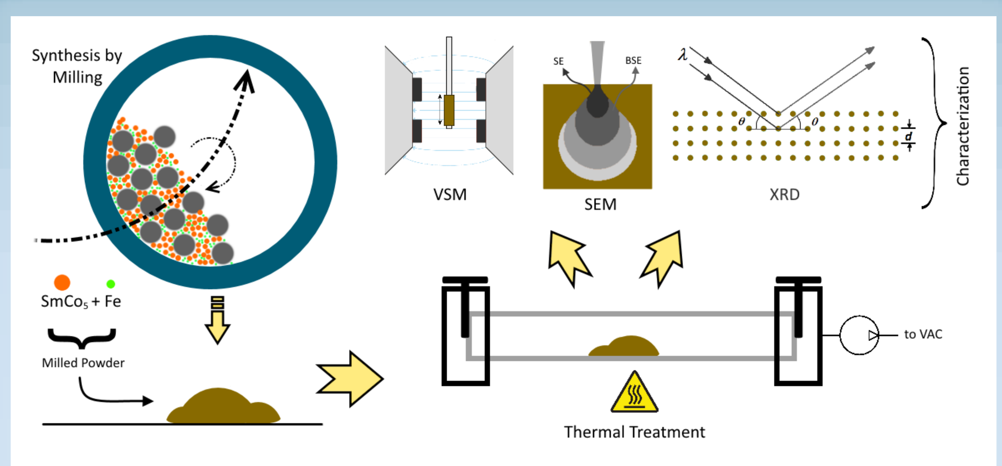

Fig.2 Schematic of the steps involved in synthesis and characterization.

Synthesis and Characterization:

This study uses 10 wt% Fe {iron} as the soft-magnetic phase fraction and 90 wt% $SmCo_5$ {samarium-cobalt 1:5} as the hard-magnetic phase. This hard-phase, namely $SmCo_5$, is either virgin (V), or recycled (R) from production magnets. We select high-energy planetary ball-milling to create homogenous milled product which is further thermally treated specifically to relax structural stress, improve the crystallinity of the hard magnetic phase, while also minimizing Fe grain-growth. DSC results help determine the thermal treatment. XRD is used to estimate crystallite sizes and confirm the phases present. SEM investigation (SE/BS) helps determine powder quality (size and morphology). With a VSM, we probe the magnetic properties of powders. All air-sensitive work is carried out in an enclosed argon-atmosphere.

Results and Conclusion:

Drawing from prior work [4], we selected 2, 4, and 6 h high-energy ball-milling to synthesize a fine homogenous milled product. This powder has 10 wt% Fe ideally embedded in a matrix of $SmCo_5$ – with a fine microstructure.

Preliminary DSC confirmed known transitions for Fe, and $SmCo_5$ suggesting thermal treatment in the 400 to 600°C temperature range. Similarly, XRD showed the presence of $SmCo_5$ and Fe with small quantities of Sm_2Co_7 and Sm_2Co_{17} and evidenced a diffuse structure. SEM investigation showed a homogenous powder consisting of aggregates with highly disrupted fine flake-like features.

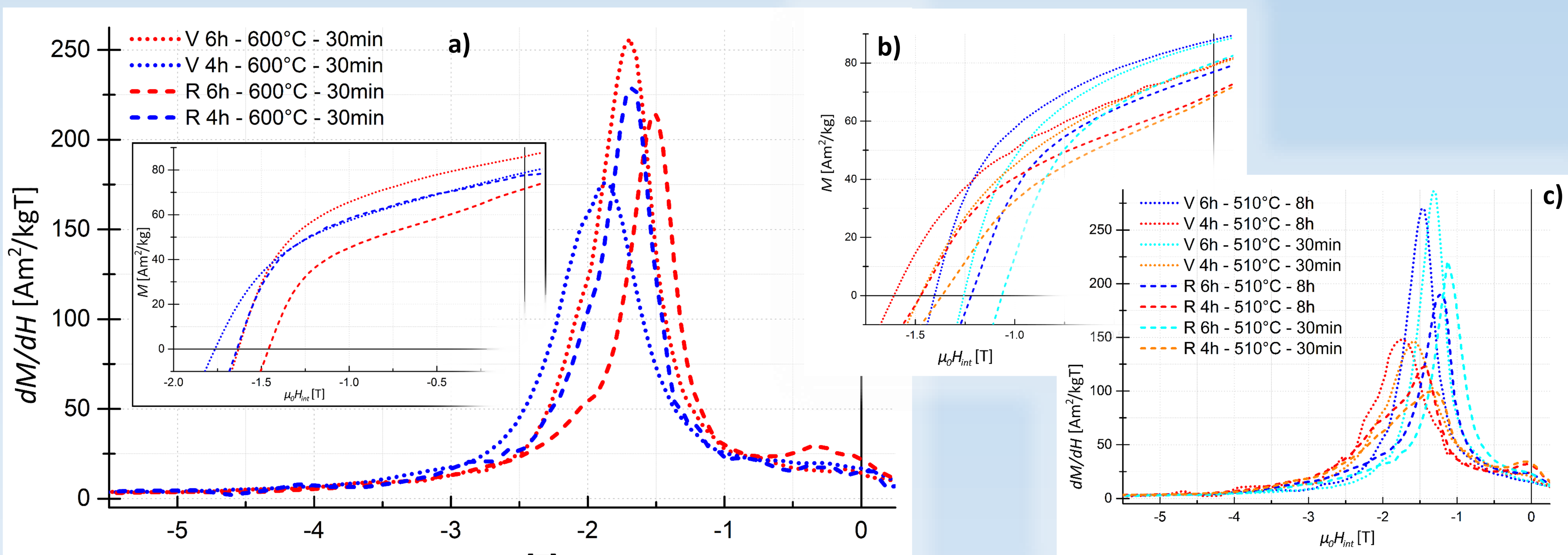

Fig.3 Red-tone curves indicate 6 h of milling, blue-tone is for 4h. Dotted curves are for virgin materials, and recycled materials are shown with dashed curves. In the derivative of M curve, (a) shows the highly coupled behavior of powders annealed at 600 °C, inset contains demagnetization curves; (b) shares legend with (c) and they show powders evolving high remanence at the cost of coercivity; at the same time, the difference between the virgin and recycled material is apparent.

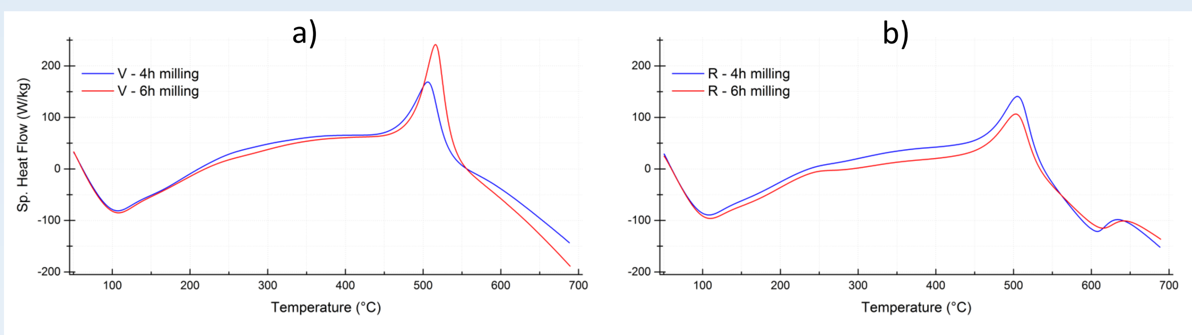

Fig.4 DSC curves helped identify zones and times of optimum annealing. (a) and (b) show transitions for virgin and recycled materials respectively.

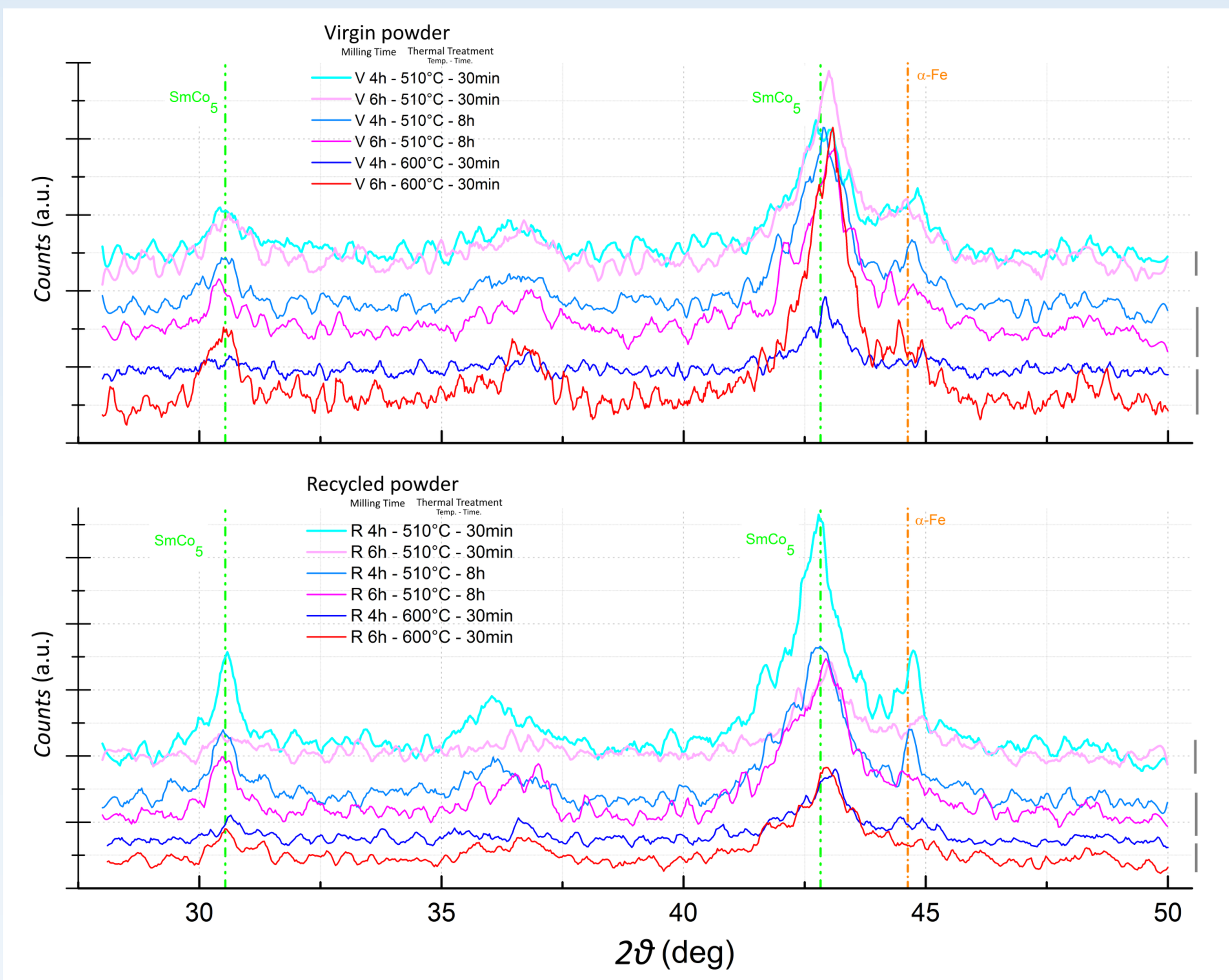

Fig.5 XRD plots obtained showed a very fine microstructure (post annealing) with $SmCo_5$ recrystallization being dominant but different for Virgin and Recycled powder. Structural difference between the two precursor materials is apparent. Most intense peaks have been identified to their phases. The plots contain

Magnetic properties depend strongly on milling time, and the nature of the precursors. In our study, the virgin $SmCo_5$ powder consistently performed better than the recycled powder. Milling for 2 h and thermal treatment at 420 °C did not develop desirable magnetic properties. Barring these, there is a compromise between high remanence and coercivity.

The virgin precursor-mix milled for 6 h and thermally treated at 600 °C for 0.5 h showed the highest remanence ($M_r \approx 86$ Am²/kg) with high coercivity ($\mu_0H_{int} \approx 1.63$ T). Thermal treatment for the same milled product at 510 °C results in the same remanence but with lower coercivity – lower still for shorter treatments. Saturation magnetization (M_s) hovers near 110 Am²/kg.

The recycled precursor-mix milled for 4 h and thermally treated at 600 °C for 0.5 h showed the highest remanence ($M_r \approx 78$ Am²/kg) with high coercivity ($\mu_0H_{int} \approx 1.65$ T). The recycled precursor-mix milled for 6 h and thermally treated at 510 °C also show high remanence ($M_r \approx$ 80-68 Am²/kg) but have lower coercivity ($\mu_0H_{int} \approx$ 1.05-1.47 T). Saturation magnetization (M_s) hovers near 100 Am²/kg.

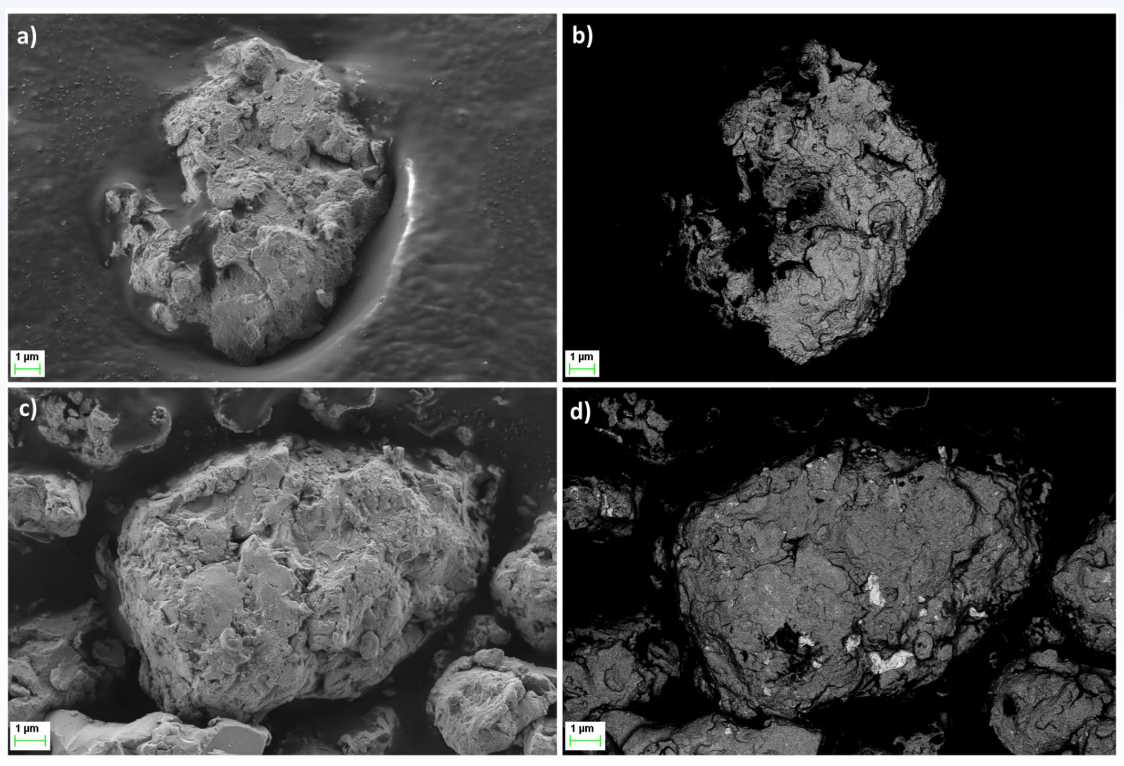

Fig.6 Aggregates are formed during dry milling: very fine flake-like particles clumped together forming the larger micron sized particle. (a) and (b) are respectively SE and BS images for the Virgin powder and (c) and (d) similarly are for Recycled powder – both milled for 4h.

The demagnetization curves show high degree of coupling and corresponding dM/dH curves confirm single phase behavior. Theoretical energy product $(BH)_{max}$ (calculated using an estimated theoretical density) is ≥ 100 kJ/m³. Milling time, and therefore degree-of-mixing, largely dictates the achievable coupling while the thermal treatment influences remanence and coercivity, as structure develops. Discrepancy in the results for virgin and recycled materials is now leading us to look at the properties of the precursors; especially hardness, and the chemistry and structure post thermal treatment. The preparation of the recycled material is another factor that may be contributive to the difference.

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Project website: http://etn-demeter.eu/

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European Commission

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