

COMBINED MÖSSBAUER SPECTROMETRY AND ATOM PROBE TOMOGRAPHY INVESTIGATION OF MECHANICALLY MILLED RARE EARTH / TRANSITION METAL POWDERS

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ABSTRACT. The microstructure of mechanically milled Nd₂Fe₁₄B, SmCo₅/Fe and SmCo₃Cu₂/α-Fe alloys has been investigated by Mössbauer spectrometry and atom probe tomography. The structural changes that occur upon milling and subsequent annealing were characterised at a nanometer scale. Milling promotes intermixing of the elements and the formation of nanograins. In Nd-Fe-B powders, the Nd₂Fe₁₄B phase is transformed into α-Fe and an amorphous Nd-Fe-B phase. In SmCo₅/Fe powders, Fe/Co intermixing occurs, leading to the formation of Sm(Co,Fe)₅ and α-Fe(Co) phases. In SmCo₃Cu₂/Fe powders, although Sm is partly oxidised, Fe/Co intermixing occurs as well, leading to the formation of α-Fe(Co) nanograins surrounded by Sm(Co,Cu,Fe)₅ intergranular regions. The combined Mössbauer and atom probe tomography investigation of rare earth / transition metal powders thus allows revealing the effect of mechanical milling and subsequent annealing on the nanostructure of the powders. These results are helpful for the understanding of the magnetic properties of the powders.

Keywords: rare earths-transition metals powders, Mössbauer spectrometry, atom probe tomography

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1. INTRODUCTION

Up today, hard magnetic materials based on rare earth / transition metal phases have the best magnetic properties among the permanent magnets materials. With the aim to increase further the magnetic properties of these permanent magnet materials, it is possible to reduce the dimensions of the grains down to the nanometer scale. Such a grain size reduction can be achieved by mechanical milling and subsequent annealing.

Another way to improve the magnetic properties of rare earth / transition metal materials is to obtain nanocomposites consisting of an intimate mixture of two magnetic phases, one soft (Fe_3B , $\alpha\text{-Fe}$...), and one hard ($\text{Nd}_2\text{Fe}_{14}\text{B}$, SmCo_5 ...). Such a nanostructure combines the high coercivity of the hard magnetic phase with the high magnetisation of the soft magnetic phase, the two phases being coupled by exchange [1,2].

These so-called “spring magnets” can lead to energy products $(\text{BH})_{\text{max}}$ higher than those achieved in the existing sintered magnets [3]. However, the role of the microstructure in the spring mechanism is not well understood [1,3-6]. Several studies were performed in view of obtaining spring magnet-type magnetic materials by mechanical alloying (MA) or mechanical milling (MM) [7–14]. Since the discovery of intergrain exchange coupling, MA has become a widely used preparation technique to obtain nanocrystalline structures of magnetic materials.

With the aim at contributing to the understanding of intergrain exchange-coupling, a structural characterization at a nanometer scale of hard/soft magnetic powders is required. From this point of view, combined Mössbauer spectrometry and atom probe tomography investigations is useful.

Mössbauer spectrometry is a very useful tool for investigating structural transformations occurring during mechanical milling of rare earth / transition metal powders. Because this technique is sensitive to the environment of Fe atoms, it allows following accurately the evolution of iron containing phases upon milling and subsequent annealing. In each case, the Mössbauer investigation was completed by atom probe tomography analysis. Atom probe tomography (APT) is a high resolution analytical microscope which provides a 3D mapping at the atomic scale of the spatial distribution of atoms in the analysed specimen [15-17]. It is particularly well suited to identify small clusters and analyse chemical heterogeneities.

Here we present the results of combined Mössbauer spectrometry and atom probe tomography investigations of mechanically milled $\text{Nd}_2\text{Fe}_{14}\text{B}/\text{Fe}$, SmCo_5/Fe and $\text{SmCo}_3\text{Cu}_2/\text{Fe}$ powders. Some results have been previously reported [18-23].

2. EXPERIMENTAL

Ingots with $\text{Nd}_2\text{Fe}_{14}\text{B}$, SmCo_5 and SmCo_3Cu_2 nominal compositions were prepared by induction melting under argon atmosphere. The purity of the starting elements was 99.9%.

The $\text{Nd}_2\text{Fe}_{14}\text{B}$ ingot was crushed and the resulting powder was sieved through a 500 μm sieve. The sieved powder was dry-milled under Ar atmosphere in a planetary mill for 1, 2, 4, 8 and 12h. The milling vials (with a volume of 80 ml) and balls (\varnothing 10 mm in diameter) were made of 440 C hardened steel. The ratio between the rotation speed of the disc and the relative rotation speed of the vials was $\Omega/\omega = 333/-900$ rpm with a ball-to-powder weight ratio of 10:1.

The SmCo_5 ingot was crushed into small pieces and it was subsequently mechanically milled for two hours in a high energy planetary mill under argon atmosphere. The obtained SmCo_5 powder was mixed with an Fe powder (grain size below 40 μm) in a ratio of 80 wt%(SmCo_5)/ 20 wt%(Fe), and the mixture was mechanically milled for an additional time of 8h. The mechanically milled powder was placed in a sealed evacuated silica tube and annealed at temperatures up to 650°C for 0.5h to remove the internal strains and defects introduced during milling.

The SmCo_3Cu_2 ingot was crushed into small pieces and subsequently mechanically milled for 2 h under argon atmosphere. The SmCo_3Cu_2 powder thus obtained was mixed with an iron powder (grain size below 40 μm) in a ratio of 70 wt% (SmCo_3Cu_2)/30 wt% (Fe). The mixture was mechanically milled under argon atmosphere using the high-energy planetary mill. Several milling times were used ranging from 1.5 to 9 h. In order to investigate the influence of the annealing on the structural evolution of the $\text{SmCo}_3\text{Cu}_2/\text{Fe}$ powder, samples of milled powder were sealed inside evacuated silica tubes for further heat treatments. These treatments were performed at temperatures ranging from 400 to 450°C for times ranging from 1 min to 2 h.

^{57}Fe Mössbauer spectrometry experiments were performed at room temperature in transmission geometry, using a ^{57}Co source in a rhodium matrix. The Mössbauer spectra were fitted according to a least squares method [24]. The isomer shift (relative to metallic $\alpha\text{-Fe}$ at room temperature) and hyperfine field are denoted δ and B respectively.

The APT experiments were performed with a Laser Assisted Wide Angle Tomographic Atom Probe from CAMECA. For sample preparation, a single particle with a diameter in the range 1-50 μm was mounted on a stainless steel fine tip needle with conductive epoxy glue, using an ex-situ micro-manipulator. The particle

was tip-shaped using a focused Ga ion beam (30 kV) [25]. To reduce Ga implantation and avoid structural damages, the final step of milling was performed at low acceleration voltage (2 kV). The tips were analyzed at 80 K in an ultrahigh vacuum chamber at a pressure of 10^{-9} mbar. The femtosecond laser pulse system used was an amplified ytterbium-doped laser (Amplitude System s-pulse) with a 350 fs pulse length and a 332 nm wavelength.

3. RESULTS

3.1. $Nd_2Fe_{14}B$ powder

The room temperature Mössbauer spectra of the $Nd_2Fe_{14}B$ powder before milling and milled for 2 and 12 hours are shown in figure 1. One can see a clear evolution of the Mössbauer spectrum with milling time, indicating important structural changes induced by mechanical milling.

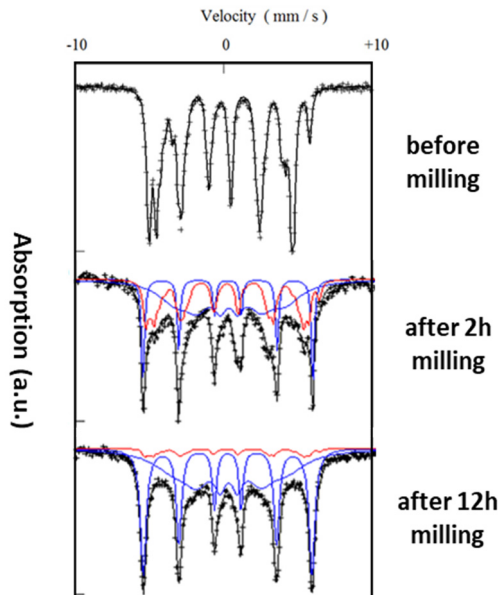


Figure 1 – Room temperature Mössbauer spectra of the Nd-Fe-B powder before milling, and after milling for 2h and 12h. In the spectra of the milled powders, the red contribution corresponds to the $Nd_2Fe_{14}B$ phase, the blue sextet contribution corresponds to the α -Fe phase and the blue large contribution corresponds to an amorphous contribution.

The spectrum of the powder before milling was fitted with the contribution of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase. The spectra of the milled powders were fitted, according to a fitting procedure previously described [26], with three contributions: the contribution of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase, the contribution of the $\alpha\text{-Fe}$ phase and, a contribution typical for an amorphous phase. As the milling time increases, the relative intensity of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase contribution decreases, as those of both $\alpha\text{-Fe}$ and amorphous phases increase. This clearly shows the disappearance of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase induced by milling and its transformation into $\alpha\text{-Fe}$ and amorphous phases.

A 3D reconstruction of a volume of the powder milled for 2 hours derived from APT analysis is shown in figure 2a. The corresponding Nd, Fe and B concentration profiles in the analyzed volume are also shown. One can clearly see the presence of three regions with different compositions:

- a region constituted of a mixture of iron, neodymium and boron (left part of the figure), corresponding to the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase also detected in the Mössbauer spectrum,

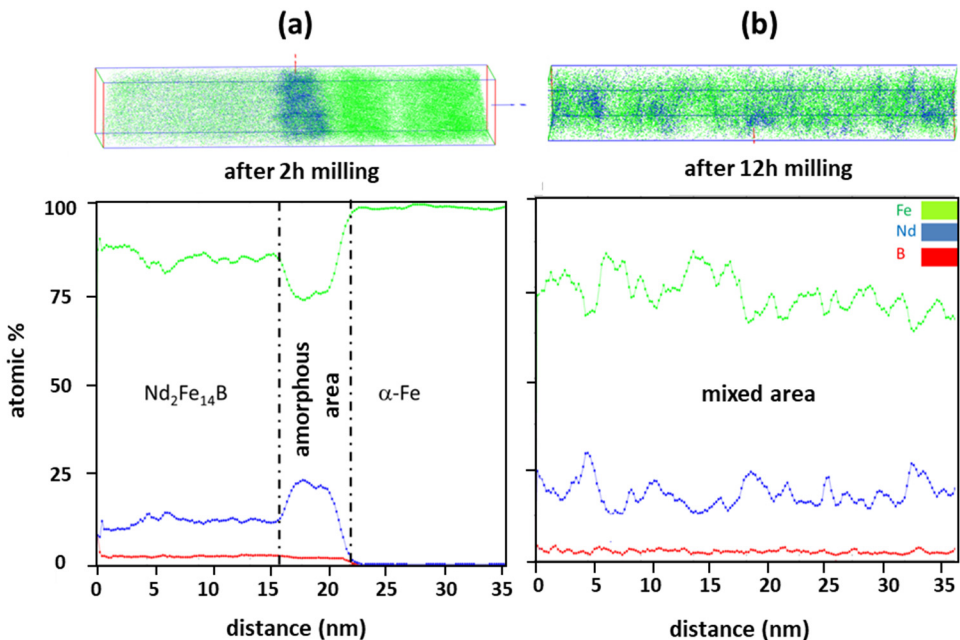


Figure 2 –3D reconstruction obtained by APT of an analysed volume of the Nd-Fe-B powder milled for 2h (a) and 12h (b) and corresponding concentration profiles.

- a neodymium enriched Nd-Fe-B region (middle part of the picture), attributed to the amorphous contribution fitted in the Mössbauer spectrum,
- a region almost entirely constituted of Fe atoms (right part of the figure), corresponding to the α -Fe Mössbauer contribution.

A 3D reconstruction of a volume of the powder milled for 12 hours derived from APT analysis is shown in figure 2b. The corresponding Nd, Fe and B concentration profiles in the analyzed volume are also shown. A so-called mixed area is observed, revealed by variations of the Fe and Nd composition at the nanometer scale. This indicates that the powder milled for 12 hours contains nanosized regions that are not pure and present diffuse interfaces. Increasing the milling time thus promotes the intermixing of Nd, Fe and B elements leading, in agreement with the Mössbauer analysis, to the almost complete disappearance of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase and to the formation of Nd-Fe-B amorphous regions and α -Fe regions, mixed at a nanometer scale.

3.2. SmCo_5/Fe powder

The room temperature Mössbauer spectra of the SmCo_5/Fe powder before milling and milled for 2 and 8 hours are shown in figure 3. An evolution of the Mössbauer spectrum with milling time is observed, related to structural changes induced by mechanical milling.

The spectrum of the powder before milling was fitted with the contribution of the α -Fe phase, in relation with the fact that before milling, all the iron atoms are present in the α -Fe phase. The spectra of the milled powders were fitted according to a fitting procedure previously described [21], with two contributions. These contributions are:

- a magnetic contribution with a hyperfine field between 33 and 35 T typical of an Fe-Co alloy, corresponding to a Fe-Co phase,
- a magnetic contribution with a mean hyperfine field typical of a rare-earth / transition metal alloy, corresponding to a Sm-Co-Fe phase.

As the milling time increases, the relative intensity of the Sm-Co-Fe phase contribution increases, as that of Fe-Co phase decreases. This shows that milling induces an intermixing of Fe and Co atoms, leading to the introduction of Co atoms in the α -Fe phase and Fe atoms in the SmCo_5 phase. These two phases are thus denoted α -Fe(Co) and $\text{Sm}(\text{Co},\text{Fe})_5$ respectively.

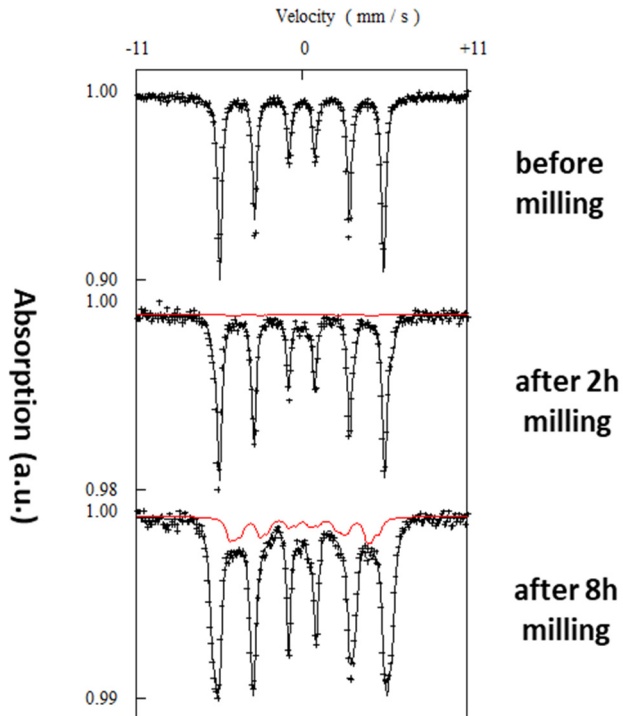


Figure 3 – Room temperature Mössbauer spectra of the SmCo_5/Fe powder before milling, and after milling for 2h and 12h. In the spectra of the milled powders, the red contribution corresponds to the $\text{Sm}(\text{Co},\text{Fe})_5$ phase.

The room temperature Mössbauer spectra of the SmCo_5/Fe powder milled for 8 hours and annealed for 0.5 hour at 500 and 550°C are shown in figure 4. As compared with the spectrum of the as-milled powder, one can see that the contribution of the Sm-Co-Fe phase increases with the annealing temperature, in agreement with an increase of the Fe/Co intermixing.

A 3D reconstruction of a volume of the powder milled for 8 hours derived from APT analysis, is shown in figure 5. The figure shows the spatial distribution of the Sm, Co, and Fe atoms in the analysed volume. A slice taken from the 3D reconstruction is shown in the figure, presenting the individual spatial distribution of each type of atoms. The Sm atoms are only detected in Sm-Co-rich regions. The regions where Sm is not present correspond to the Fe rich regions in the Fe map, being of a few nanometers in size. The Fe atoms are also present in the Sm-rich regions. The Co

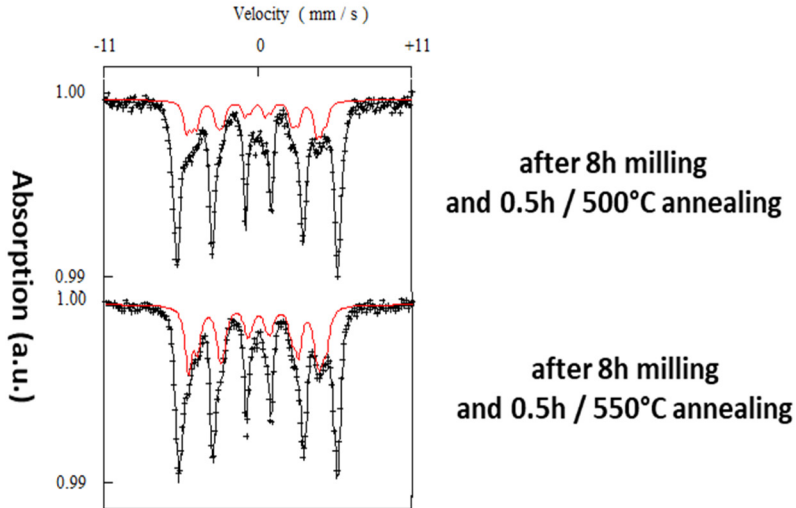


Figure 4 – Room temperature Mössbauer spectra of the SmCo_5/Fe powder after milling and annealing for 0.5h at 500 and 550°C. The red contribution corresponds to the $\text{Sm}(\text{Co,Fe})_5$ phase.

atoms are detected in both the Sm-rich and the Fe-rich regions, their concentration being higher in the Sm-rich regions. The mean composition of the Sm-rich regions is $20\pm 2\%$ Sm, $70\pm 2\%$ Co and $10\pm 2\%$ Fe, being consistent with a $\text{Sm}(\text{Co,Fe})_5$ phase. The mean composition of the Fe-rich regions is $75\pm 2\%$ Fe, and $25\pm 2\%$ Co.

These results are fully consistent with the Mössbauer analysis and confirm that mechanical milling leads to Fe/Co intermixing, leading to the presence in the as-milled powders of two phases: a $\text{Sm}(\text{Fe,Co})_5$ phase and a $\text{Fe}_{75}\text{Co}_{25}$ phase. These two phases are present as grains with a nanometer size.

A 3D reconstruction of the SmCo_5/Fe powder milled for 8h and annealed for 0.5h at 550°C is shown in figure 6. The nanoscale morphology of the annealed powder is comparable to that of the as-milled powder, as the powder contains nanosized Fe(Co) clusters embedded in the $\text{Sm}(\text{Co,Fe})_5$ phase. The mean Fe content in the $\text{Sm}(\text{Co,Fe})_5$ phase is slightly higher than the value measured in the as-milled sample, being $15\pm 2\%$. The Co content in the Fe(Co) is statistically higher after annealing amounting to $45\pm 2\%$ Co. These variations of the atomic concentrations upon annealing confirm that the Fe/Co interdiffusion is thermally activated. The increase of the Fe content in the Sm-rich regions and the increase of the Co content in the Fe-rich regions upon annealing are consistent with the results of the Mössbauer analysis.

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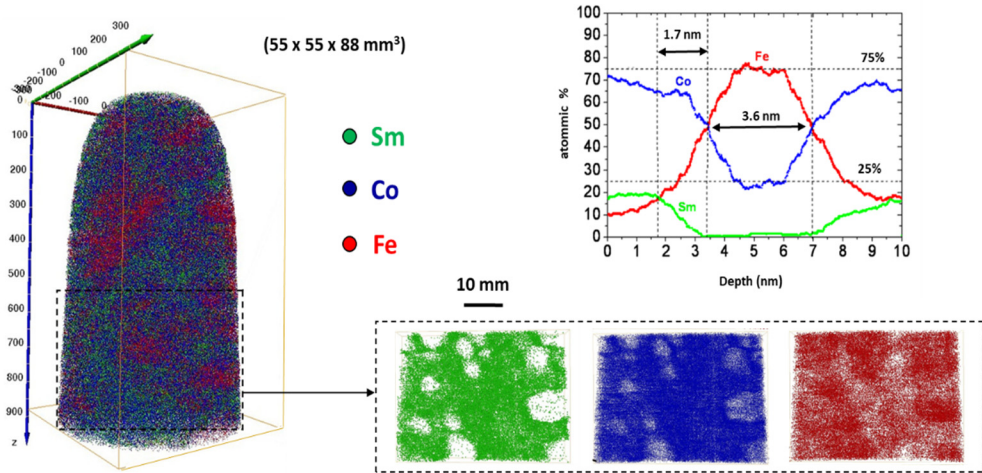


Figure 5 – 3D reconstruction obtained by APT of an analysed volume in the SmCo_5/Fe powder milled for 8h, and concentration profile through a Fe-rich region. The distribution of Sm, Co and Fe atoms in a 30 nm thick slice taken from the analysed volume is shown.

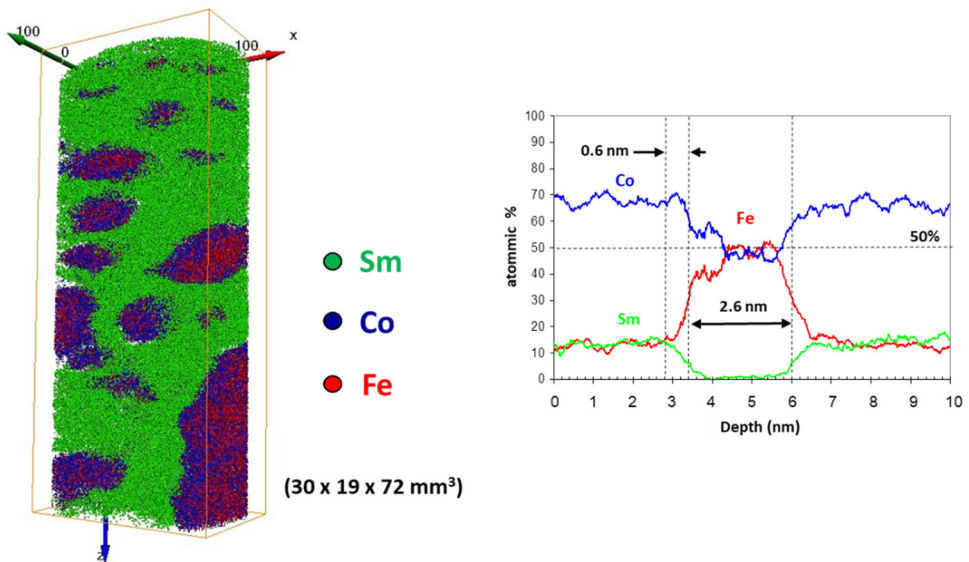


Figure 6 – 3D reconstruction obtained by APT of an analysed volume of the SmCo_5/Fe powder milled for 8h and subsequently annealed for 0.5h at 550°C and concentration profile through a Fe-rich region.

3.3. $\text{SmCo}_3\text{Cu}_2/\text{Fe}$ powder

The room temperature Mössbauer spectra of the $\text{SmCo}_3\text{Cu}_2/\text{Fe}$ powder before milling and milled for 9 hours are shown in figure 7. The room temperature spectrum of the powder milled for 9 hours and subsequently annealed for 0.5 hours at 450°C is shown in the same figure.

The spectrum of the powder before milling was fitted with the contribution of the $\alpha\text{-Fe}$ phase, in relation with the fact that before milling, all the iron atoms are present in the $\alpha\text{-Fe}$ phase. The spectra of the milled powders were fitted with two contributions:

- a magnetic contribution with a hyperfine field between 33 and 35 T typical of an Fe-Co alloy, corresponding to a Fe-Co phase,
- a magnetic contribution with a mean hyperfine field typical of a rare-earth / transition metal alloy, corresponding to a Sm-Co-Cu-Fe phase.

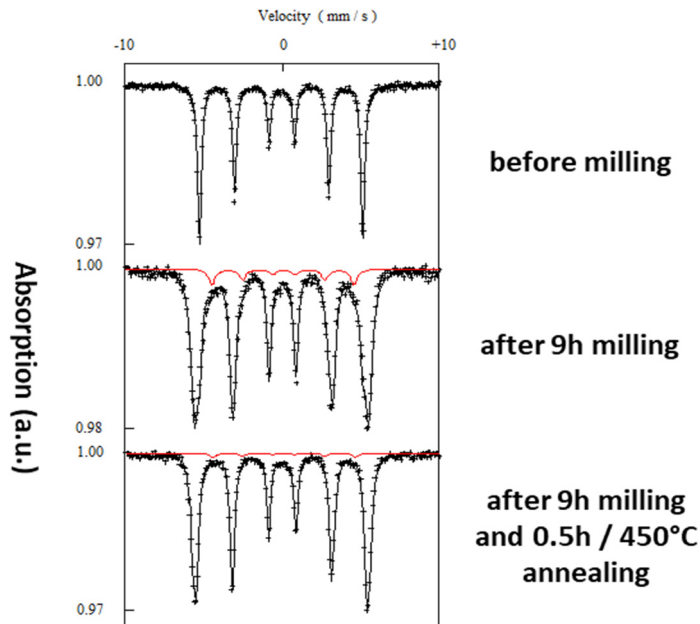


Figure 7 – Room temperature Mössbauer spectra of the $\text{SmCo}_3\text{Cu}_2/\text{Fe}$ powder before milling, after milling for 9h and after milling for 9h and subsequent annealing for 0.5h at 450°C . The red contribution corresponds to $\text{Sm}(\text{Co},\text{Fe},\text{Cu})$ regions.

After 9 hours milling, the relative intensity of the Sm-Co-Cu-Fe phase contribution is lower than that of the Sm-Co-Fe phase in the SmCo_5/Fe powder after 8 hours milling. This suggests that the Fe/Co intermixing upon milling is lower in the $\text{SmCo}_3\text{Cu}_2/\text{Fe}$ powder than in the SmCo_5/Fe powder.

After annealing, the Mössbauer spectrum of the powder milled for 9 hours is mainly composed of the contribution of the Fe-Co phase, as the relative intensity of the Sm-Co-Cu-Fe phase is very small, being lower than in the as-milled powder. This indicates that only a few Fe atoms are present in the Sm-rich phase after annealing, suggesting that the Fe/Co intermixing is not as important as in the $\text{SmCo}_3\text{Cu}_2/\text{Fe}$ powders. As compared with the behaviour of the SmCo_5/Fe powder upon annealing, the behaviour of the $\text{SmCo}_3\text{Cu}_2/\text{Fe}$ powder upon milling and subsequent annealing could be related to the presence of Cu atoms.

3D reconstructions of a volume of the powder milled for 9 hours, before and after annealing for 0.5 hour at 450°C , derived from APT analysis, are shown in figure 8. Slices taken from the 3D reconstruction are shown in the figure, presenting the individual spatial distributions of Sm, Co, Cu and Fe atoms.

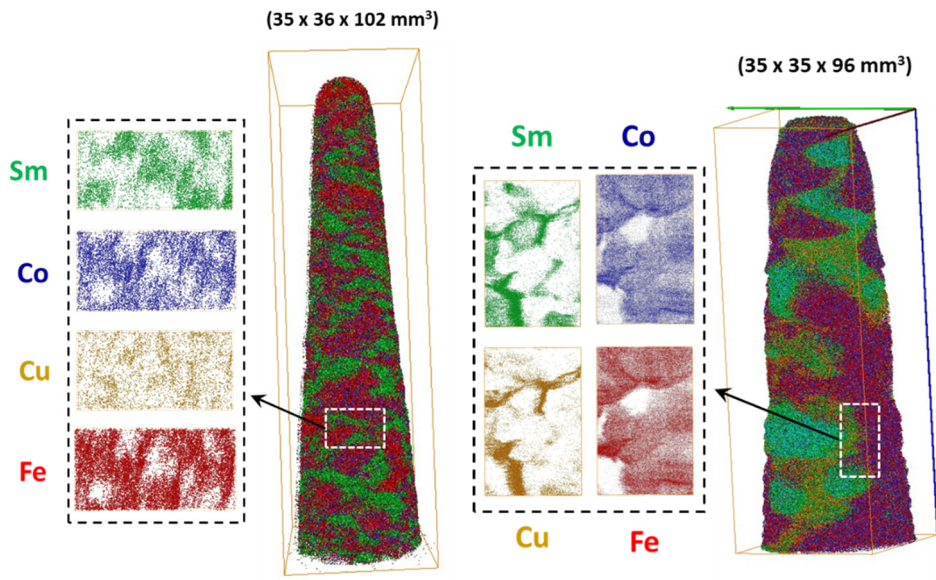


Figure 8 – 3D reconstruction obtained by APT of an analysed volume in the $\text{SmCo}_3\text{Cu}_2/\text{Fe}$ powder milled for 9h before (left) and after (right) annealing for 0.5h at 450°C .

The distributions of Sm, Co, Cu and Fe atoms in a 5 nm thick slice taken from the analysed volume are shown.

In the as-milled powder, one can observe the presence of Sm-rich regions, containing a few Co and Fe atoms, and Sm-poor regions, containing a high amount of Co and Fe atoms. Cu atoms are present in the whole analysed volume, with a concentration that is more important in Sm-rich regions than in Sm-poor regions.

In the annealed powder, three different regions are observed: Sm-rich regions with almost no Co, Fe or Cu atoms, Sm-poor regions with a high amount of Co and Fe atoms, and Sm-Co-Cu-Fe regions that seem to correspond to intergranular regions. The Sm-rich regions could correspond to Sm oxides.

These results are in agreement with Mössbauer analysis, showing that milling induces Fe/Co intermixing, leading to the formation of a Fe-Co phase and Sm-Co-Cu-Fe regions. However, the proportion of the Sm-Co-Cu-Fe phase is low, as Sm-rich regions almost pure in Sm are observed that could correspond to Sm oxides. Annealing promotes the formation of Fe-Co nanograins surrounded by intergranular Sm-Co-Cu-Fe regions.

4. CONCLUSION

The combined Mössbauer and APT investigation of rare earth / transition metal powders allows revealing the effect of mechanical milling and subsequent annealing on the nanostructure of the powders.

Milling $\text{Nd}_2\text{Fe}_{14}\text{B}$ powder leads to the formation of nanosized regions that are not pure and present diffuse interfaces. The intermixing of Nd, Fe and B elements is promoted. Nd-Fe-B amorphous regions and α -Fe regions, mixed at a nanometer scale, are formed.

Milling SmCo_5/Fe powders induces Fe/Co intermixing, and leads to the formation of $\text{Sm}(\text{Fe},\text{Co})_5$ and α -Fe(Co) phases that are present in grains with a nanometer size. Upon annealing, the Fe/Co interdiffusion is thermally activated, inducing an increase of both the Fe content in the $\text{Sm}(\text{Fe},\text{Co})_5$ phase and the Co content in the α -Fe(Co) phases.

Milling $\text{SmCo}_3\text{Cu}_2/\text{Fe}$ powders induces Fe/Co intermixing, and leads to the formation of a Fe-Co phase, and Sm-Co-Cu-Fe regions. However, the proportion of the Sm-Co-Cu-Fe phase is very low, as Sm-rich regions almost pure in Sm are present as well, that could correspond to Sm oxides. Annealing promotes the formation of Fe-Co nanograins surrounded by intergranular Sm-Co-Cu-Fe regions.

These results show that the combined Mössbauer spectrometry and atom probe tomography investigation of mechanically milled rare earth / transition metal powders allows obtaining both structural and chemical information at a nanometer scale that is thus useful for the understanding of magnetic interactions between nanograins.

REFERENCES

- [1] E.E. Fullerton, J.S. Jiang, M. Grimsditch, C.H. Sowers, S.D. Bader, *Physical Review B* 58 (1998) 12193
- [2] E.E. Fullerton, J.S. Jiang, S.D. Bader, *Journal of Magnetism and Magnetic Materials* 200 (1999) 392
- [3] E.F. Kneller, R. Hawig, *IEEE Transaction on Magnetics* 27 (1991) 3588
- [4] R. Skomski, *Journal of Applied Physics* 76 (1994) 7059
- [5] R. Skomski, *Journal of Physics Condensed Matter* 15 (2003) R841
- [6] R. Skomski, J.M.D. Coey, *Permanent Magnetism*, Institute of Physics Publishing, Bristol
- [7] J. Roskilde, J. Petzold, *Journal of Magnetism and Magnetic Materials* 242–245 (2002) 84
- [8] P.A.P. Wendhausen, B. Gebel, D. Eckert, K.H. Müller, *Journal of Applied Physics* 75 (1994) 6019
- [9] M. Venkatesan, C. Jiang, J.M.D. Coey, *Journal of Magnetism and Magnetic Materials* 242–245 (2002) 1350
- [10] K. O'Donnell, J.M.D. Coey, *Journal of Applied Physics* 81 (1997) 6311
- [11] L. Wei, W. Qun, X.K. Sun, Z. Xin-Guo, Z. Tong, Z. Zhi-Dong, Y.C. Chuang, *Journal of Magnetism and Magnetic Materials* 131 (1994) 413
- [12] J.X. Zhang, L. Bessais, C. Djega-Mariadassou, E. Leroy, A. Percheron-Guegan, Y. Champion, *Applied Physics Letters* 80 (2002) 1960
- [13] D.L. Leslie-Pelecky, R.L. Schalek, *Physical Review B* 59 (1999) 457
- [14] V. Neu, L. Schultz, *Journal of Applied Physics* 90 (2001) 1540
- [15] A. Cerezo, P.H. Clifton, A. Gombert, G.D.W. Smith, *Ultramicroscopy* 107 (2007) 720–725.
- [16] D. Blavette, B. Deconihout, A. Bostel, J.-M. Sarrau, M. Bouet, A. Menand, *Review of Scientific Instruments* 64 (1993) 2911–2919
- [17] M.K. Miller, R.G. Forbes, *Materials Characterization* 60 (2009) 461–469
- [18] N. Maât, R. Lardé, V. Nachbaur, J.-M. Le Breton, O. Isnard, V. Pop, I. Chicinas, *Journal of Applied Physics* 124 (2018) 223905
- [19] R. Lardé, J.-M. Le Breton, A. Maître, D. Ledue, O. Isnard, V. Pop, I. Chicinas, *Journal of Physical Chemistry C* 117 (2013) 7801–7810
- [20] V. Pop, E. Dorolti, C. Vaju, E. Gautron, O. Isnard, J.-M. Le Breton, I. Chicinas, *Romanian Journal of Physics* 55 (2010) 127–136

- [21] J.-M. Le Breton, R. Lardé, H. Chiron, V. Pop, D. Givord, O. Isnard, I. Chicinas, *Journal of Physics D: Applied Physics* 43 (2010) 085001
- [22] V. Pop, O. Isnard, D. Givord, J.-M. Le Breton, I. Chicinas, *Journal of Optoelectronics and Advanced Materials* 10 (2008) 1872-1876
- [23] V. Pop, O. Isnard, I. Chicinas, D. Givord, J.-M. Le Breton, *Journal of Optoelectronics and Advanced Materials* 8 (2006) 494-500
- [24] J. Teillet, F. Varret (1983) unpublished MOSFIT program
- [25] K. Thompson, D. Lawrence, D.J. Larson, J.D. Olson, T.F. Kelly, B. Gorman, *Ultramicroscopy* 107 (2007) 131–139
- [26] G. Khélifati, J.-M. Le Breton, L. Aymard, J. Teillet, *Journal of Magnetism and Magnetic Materials* 218 (2000) 42–48