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Microstructural features of strip-cast $Nd(Fe,Mo)_{12}$ based alloys

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$Nd(Fe,Mo)_{12}$ compounds have been considered as promising candidates for next generation permanent magnet materials because of their excellent intrinsic hard magnetic properties with a minimum usage of rare earth elements. However, achieving high coercivity in anisotropic microstructure is a big challenge, which hinders their practical applications. In this work we present the microstructural study of the strip-cast $Nd(Fe,Mo)_{12}$-based alloy. Detailed microstructure characterizations using transmission electron microscopy (TEM) indicate that addition of Cu leads to sub-micron size 1-12 magnetic grains with Nd and Cu segregated at grain boundaries. This work provides an evidence of the issues of the microstructure of these alloys for the development of the appropriate microstructure conducting to the high coercivity in the 1-12 alloys.

***Index Terms*—magnets, ThMn12-type compounds, coercivity, twin boundaries, microstructure**

I. Introduction

High performance permanent magnets (PM) are nowadays indispensable materials for green energy transition technologies as well as consumer electronics, factory automation equipment, medical instruments and therapeutics. Discovered in the early 1980s, NdFeB permanent magnets (PM) attain the highest performances with an energy product larger than 300 kJ/m^3 making them the strongest permanent magnets commercially available nowadays, with coercivities as close as 2 T [1].

Intermetallic RE-Fe_{12} compounds, based on the tetragonal $ThMn_{12}$ crystal structure, are the most promising candidates for replacing the NdFeB PM, thanks to their relatively high Curie temperatures, saturation magnetization and magnetocrystalline anisotropy. The tetragonal $ThMn_{12}$ structure of the 1-12 compounds can be stabilized thanks to the substitution of Fe by stabilizing elements like Ti, V, Cr, Mo, W or Si. Neodymium-based 1-12 compounds have usually a planar anisotropy and are not suitable for permanent magnets. Insertion of light elements, like B, C or N, will turn the planar anisotropy to easy axis and will enhance both the magnetocristalline anisotropy and Curie temperature [2].

$Nd(Fe,Mo)_{12}N$-based permanent magnets have been considered as one of the good candidates for permanent magnet applications due to their good intrinsic magnetic properties with Curie temperature T_C about 320°C, high saturation magnetization M_s up to 1T and a large magnetocrystalline anisotropy H_A up to 11T [3]. Even if the intrinsic magnetic propeties are relatevly high, at the moment coercivity is still low for permanent magnet application. Single-phase material and proper nitrogenation is a prerequisite to increase the intrinsic magnetic properties. Besides this, the relation between microstructure and the extrinsic magnetic properties are an important factor for the development the permanent magnet's performances.

A possible explanation for the low coercivity of $Nd(Fe,Mo)_{12}N$ powders may be the absence of a non-magnetic grain boundary phase which magnetically decouples adjacent grains, as found in classical $Nd_2Fe_{14}B$-type sintered magnets. Another reason might be the presence of macroscopic and microscopic defects. Twin boundaries have been already reported in the literature in the $Sm(Fe,Ti)_{12}$ [4] or $Nd(Fe,Ti)_{12}$ [5] based materials.

In the present work, we investigate the particular microstructure of the $Nd(Fe,Mo)_{12}$-based strip cast alloys. Structural and microstructural features can explain the extrinsic magnetic properties of the nitrogenated powders.

II. Experimental procedure

Alloy with nominal $(Nd_{0.75},Pr_{0.25})_{1.35}Fe_{10.5}Mo_{1.5}Cu_{0.1}$ wt.% (called $Nd(Fe,Mo)_{12}Cu$ hereinafter) composition was prepared by strip-casting (SC) technique. As the decomposition of the $Nd(Fe,Mo)_{12}$ nitride begins at 600°C [6], Cu have been added to this alloy to: (i) allow the formation of a non magnetic Nd-Cu phase at grains boundaries and (ii) facilitate the densification of the powders at low temperatures. The crystallographic structure was characterized by X-ray powder diffraction (XRD), with Cu $K_{\alpha 1}$ radiation. Microstructure observation at submicron scale was performed by transmission electron microscopy. Bright Field (BF) Scanning Transmission electron microscopy (S-TEM) investigations were conducted with a Thermo-Fisher Osiris Tecnai instrument, equipped with a X-FEG source and operating at 200 kV. Energy dispersive X-ray spectroscopy (EDX) was employed in STEM mode thanks to the Super-X EDX system. EDX hyper-maps were acquired with Esprit 1.9 software (Bruker). Crystal orientation was investigated thanks to Automated Crystallographic Orientation Mapping (ACOM) [7]. TEM sample was lifted out by focused ion beam (Zeiss FIB-550) and placed on a Mo-grid.

III. Results

Fig. 1 presents the XRD pattern of the $Nd(Fe,Mo)_{12}Cu$ powder showing three different phases: 95.5% of $NdFe_{10.5}Mo_{1.5}$, 3.5% of α-Fe and 0.5 % of Nd. Cu is not detected at this scale of analysis, probably due to the low level of crystallization or very small size of Cu containing phases.

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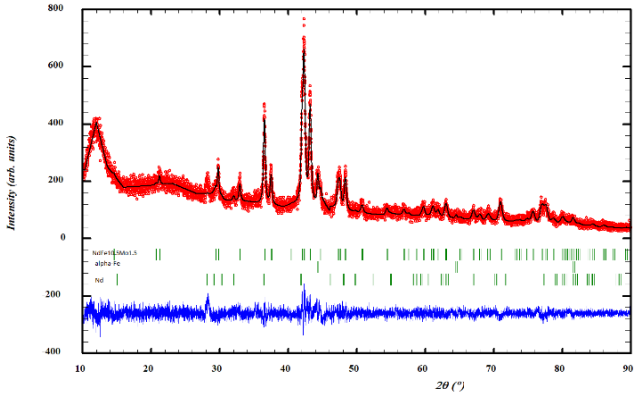

Fig. 1. XRD pattern of the $Nd(Fe,Mo)_{12}Cu$ powders.

The observation of TEM sample in BF-STEM mode reveals a microstructure constituted of fine grains, between 50 and 1000 nm as can be observed in Fig. 2.

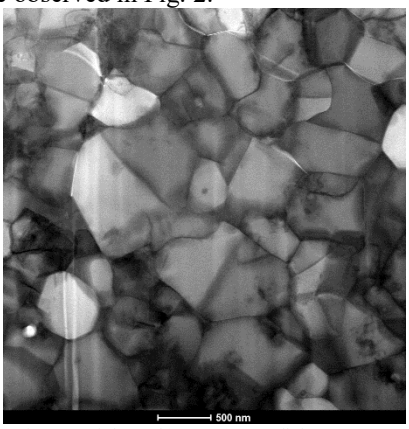

Fig. 2. BF-STEM image of the $Nd(Fe,Mo)_{12}Cu$ flakes.

EDX maps show non-uniform grain boundaries between the $Nd(Fe,Mo)_{12}$ magnetic grains (Fig. 3). Some of them are Nd and Cu segregated however some other do not present Nd or Cu enrichment. The crystal orientation analysis thanks to ACOM-TEM confirms the presence of twins in the grains. The inverse pole figure (IPF) along z axis and the misorientation angle profile across the twin boundary (Fig. 4) show an angle of approximately 58° on either side of the twin boundary plane corresponding to {011}-type plane. This microstructure is not favorable to the coercivity development, as the magnetic grains are magnetically coupled to each other.

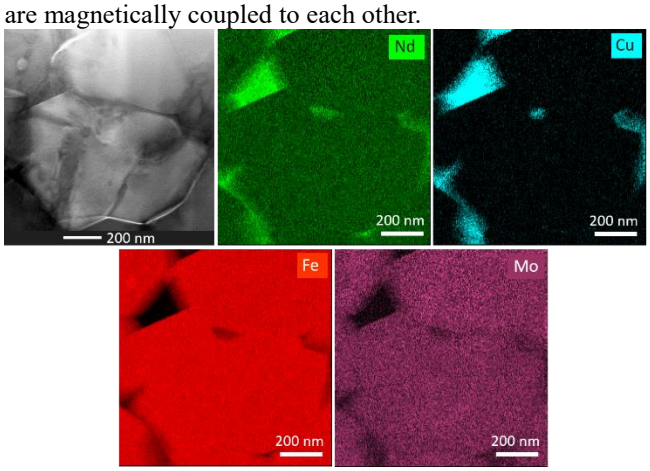

Fig. 3. Nd, Fe, Mo and Cu EDX maps of the $Nd(Fe,Mo)_{12}Cu$ flakes

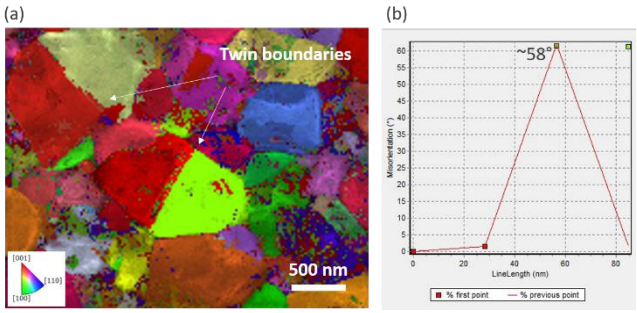

Fig. 4. Twin boundaries. (a) IPF-z (normal axis to the sample surface). (b) Misorientation angle profile across twin boundary.

IV. Conclusion

Detailed structural and microstructural characterizations in Nd(Fe,Mo) based alloys obtained by strip casting have shown the presence of twins, also highlighted in other RE-Fe12-based compounds [4], [5]. Even if it seems that this is a generic feature of these alloys, at our knowledge this is the first published evidence of twins in the $Nd(Fe,Mo)_{12}$ system. The presence of twin boundaries hinders the development of a proper microstructure necessary to achieve an efficient magnetic decoupling of the grains. Detailed analysis of the twin boundary should be perform to highlight potential segregation of elements in twin plane as already observed in literature [4] and implement strategies to modify its magnetic characteristics and countered the damaging effect of twin boundary on the coercivity.

Acknowledgment

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