

Enhancement of thermal stability of $\text{Sm}(\text{Fe-Co})_{12}\text{-B}$ thin films by cap layer deposition and post-annealing

Y. Mori, S. Nakatsuka, M. Doi, and T. Shima

Graduate School of Engineering, Tohoku Gakuin University
Sendai, Japan



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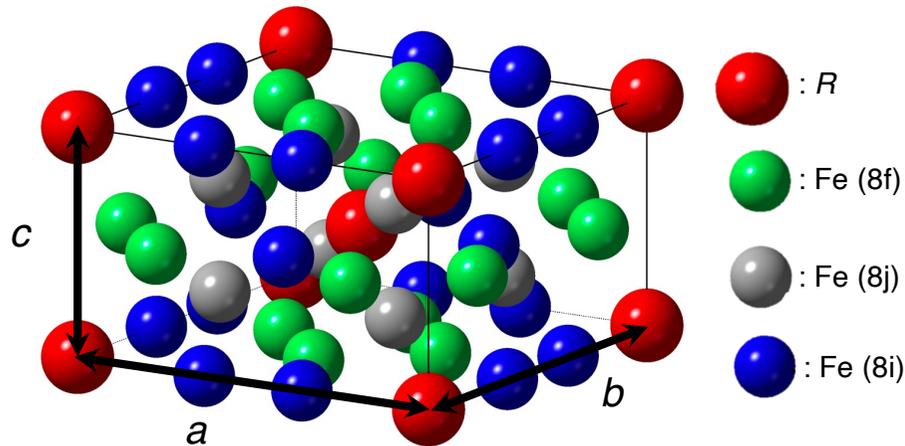
Introduction: Sm-Fe based compounds and stabilizing elements

Typical Sm-Fe-based compounds

Chemical composition	SmFe ₅	Sm ₂ Fe ₁₇	SmFe ₁₂
Crystal structure	CaCu ₅ -type	Th ₂ Zn ₁₇ -type	ThMn ₁₂ -type
Fe content (at.%)	83.3	89.5	92.3
Phase stability	Unstable	Stable	Unstable

S. Saito *et al.*, *J. Alloys Compd.*, **929** (2022) 167280.

ThMn₁₂-type crystal structure



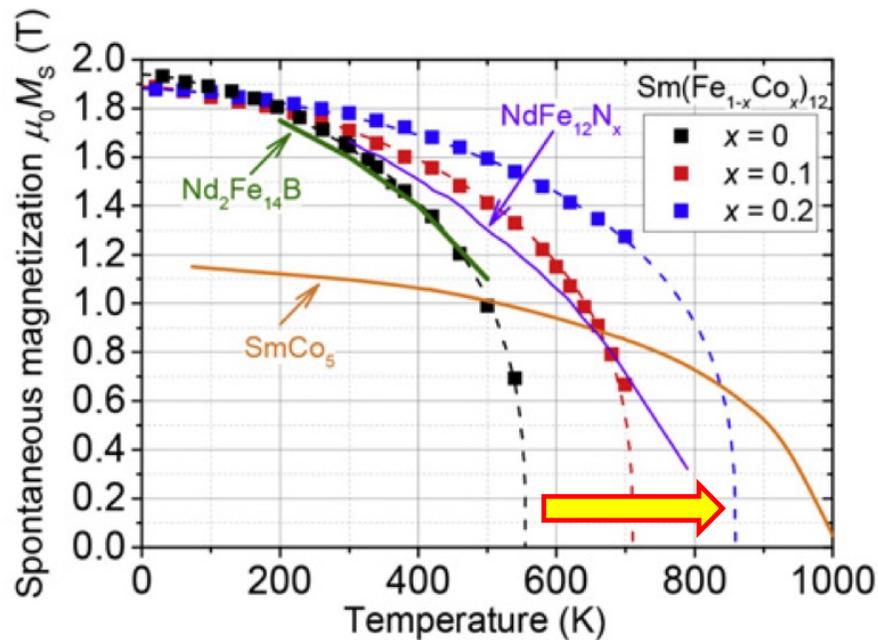
Phase stabilizing elements of SmFe₁₂

Elements	Atomic radius (nm)	Substitution site
Ti	0.147	8i
V	0.134	8i
Cr	0.128	8i, 8j, 8f
Mo	0.139	8i
Al	0.143	8j, 8f
Si	0.132	8j, 8f
Mn	0.127	8i, 8j, 8f
W	0.139	
Zr	0.16	2a
Co	0.125	8j, 8f
Nb	0.146	8i
Ta	0.146	8i
Re	0.137	
Fe	0.126	-
Sm	0.18	-

Y. K. Takahashi *et al.*, *Sci. Technol. Adv. Mater.*, **22(1)** (2021) 449-460.

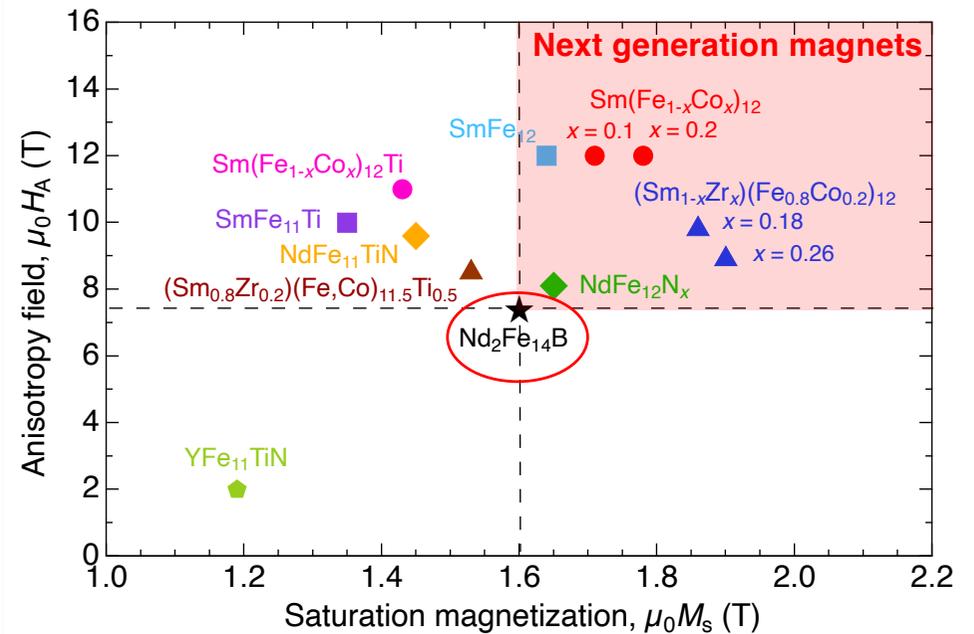
RFe₁₂ compounds: Candidate for next generation permanent magnet

$\mu_0 M_s$ -T for various magnetic materials



Y. Hirayama *et al.*, *Scr. Mater.*, **138** (2017) 62-65.

$\mu_0 M_s$ vs $\mu_0 H_A$ for various RFe₁₂ compounds

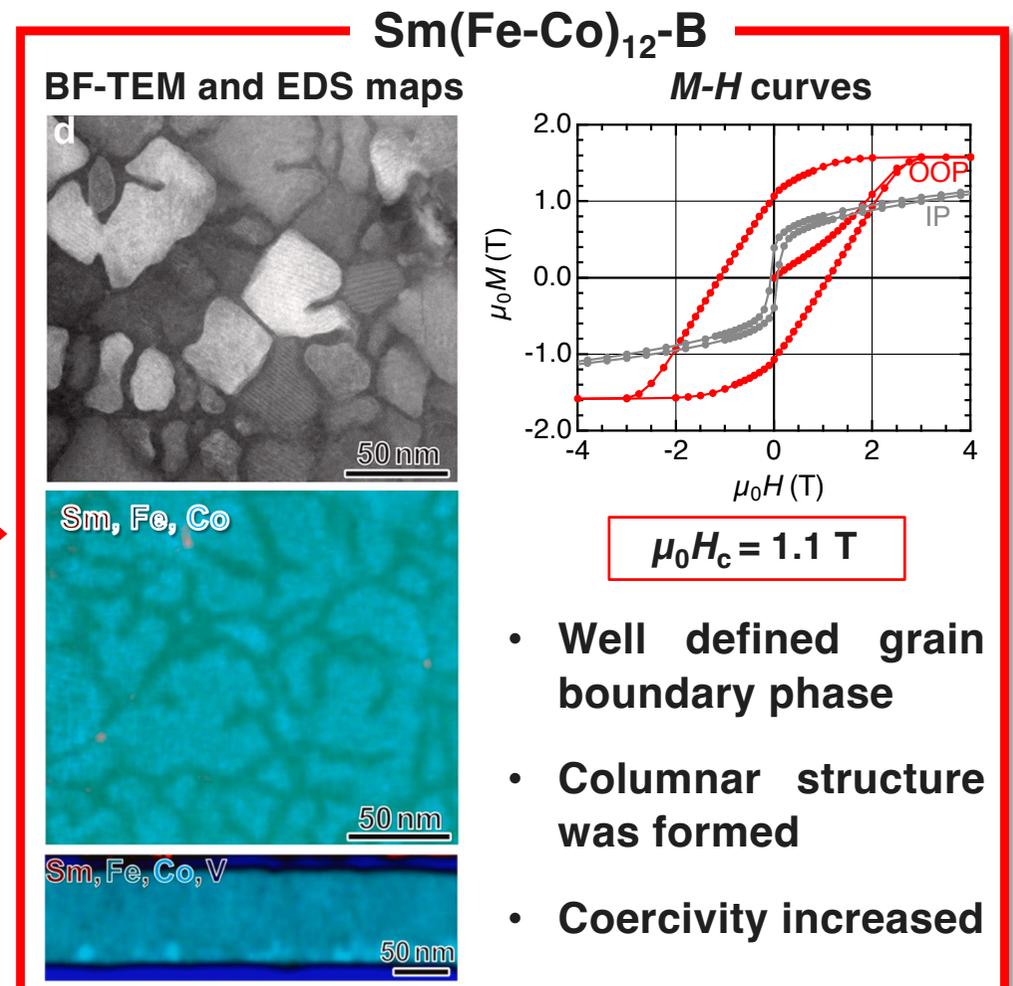
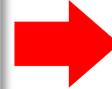
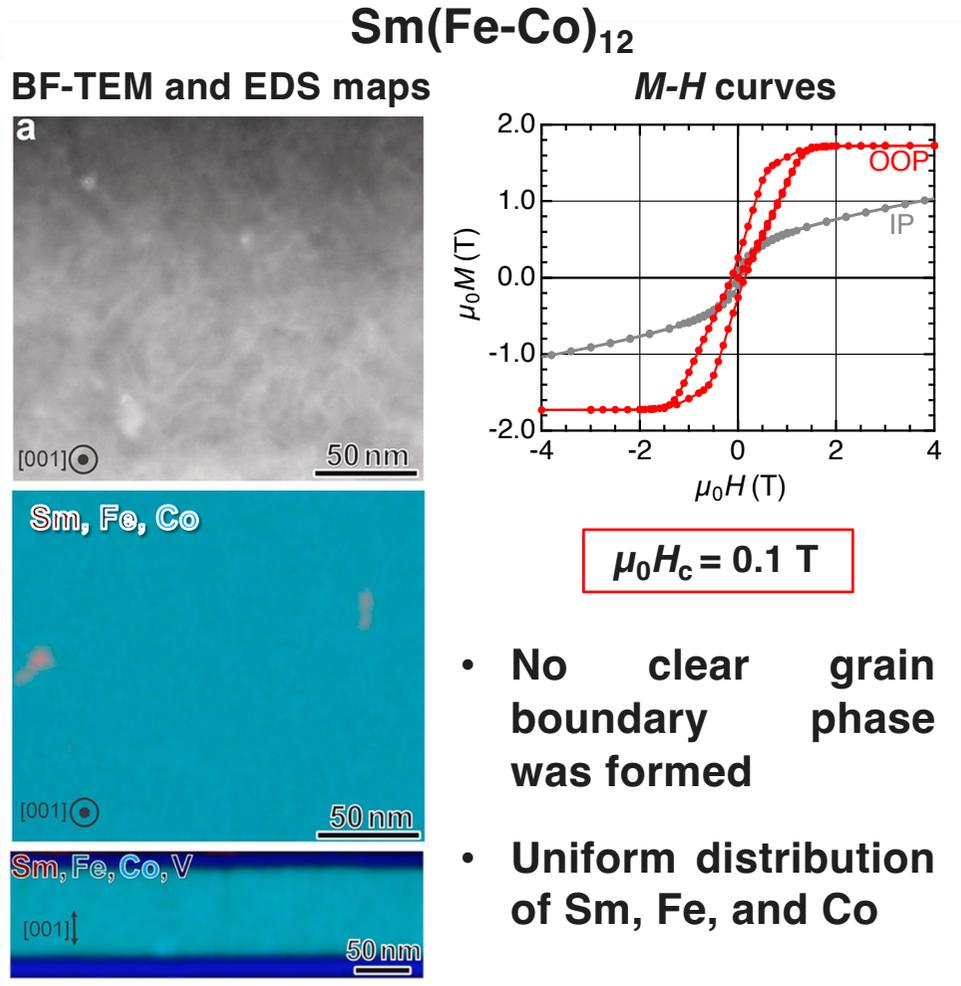


P. Tozman *et al.*, *Scr. Mater.*, **194** (2021) 113686.

- Partial substitution of Fe with Co increases the T_C of SmFe_{12} .

- $\mu_0 M_s$ and $\mu_0 H_A$ superior than $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets were realized in $\text{Sm}(\text{Fe-Co})_{12}$ compounds.

Effect of B addition for $\text{Sm}(\text{Fe-Co})_{12}$ thin films

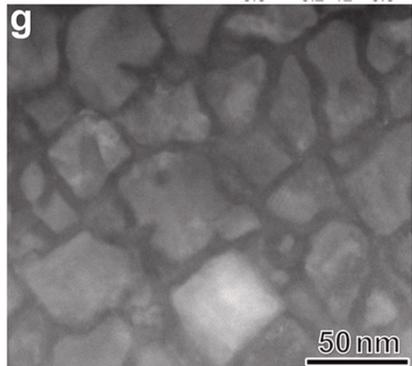


Ref: H. Sepehri-Amin *et al.*, *Scr. Mater.*, **242** (2024) 115955., M. Kambayashi *et al.*, *J. Magn. Soc. Jpn.*, **45** (2021) 66-69.

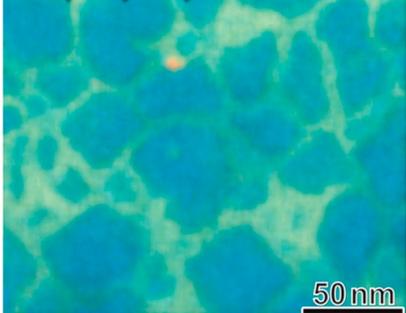
Al cap layer diffusion into GB phase in $\text{Sm}(\text{Fe-Co})_{12}\text{-B}$ thin films

$\text{Sm}(\text{Fe-Co})_{12}\text{-B}/\text{Al}$ + post annealing

BF-TEM and EDS maps



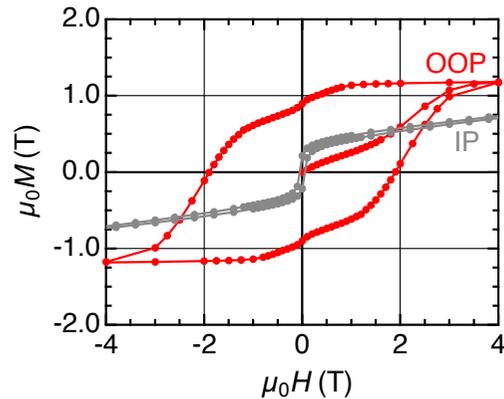
Sm, Fe, Co, Al



Sm, Fe, Co, Al, V



M-H curves



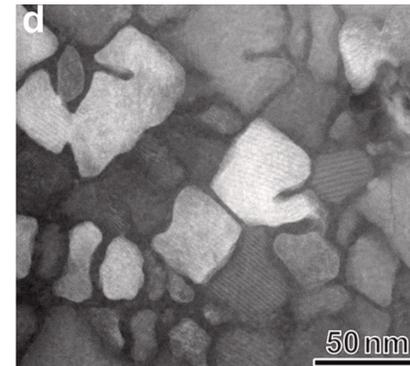
$$\mu_0 H_c = 1.8 \text{ T}$$

- Al marked with yellow was diffused down into GB phase

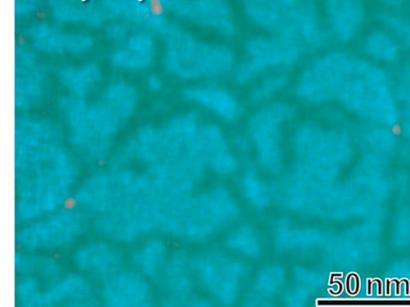
Cap layer deposition and post annealing is important to control microstructure

$\text{Sm}(\text{Fe-Co})_{12}\text{-B}$

BF-TEM and EDS maps



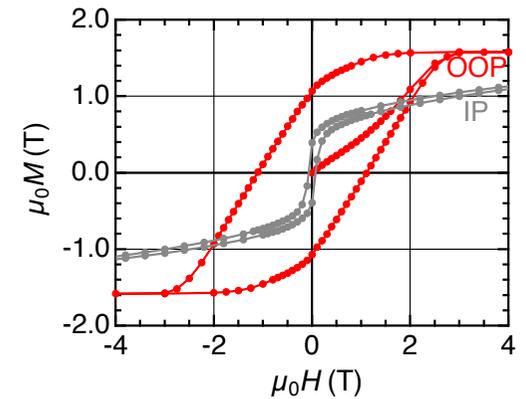
Sm, Fe, Co



Sm, Fe, Co, V



M-H curves



$$\mu_0 H_c = 1.1 \text{ T}$$

- Well defined grain boundary phase
- Columnar structure was formed
- Coercivity increased

Ref: H. Sepehri-Amin *et al.*, *Scr. Mater.*, **242** (2024) 115955., M. Kambayashi *et al.*, *J. Magn. Soc. Jpn.*, **45** (2021) 66-69.

Objective

In this study, in order to control the structure and improve the magnetic properties, stabilizing element such as Nb and Zr was selected as a capping layer material and post-annealed for the $\text{Sm}(\text{Fe-Co})_{12}\text{-B}$ thin film.

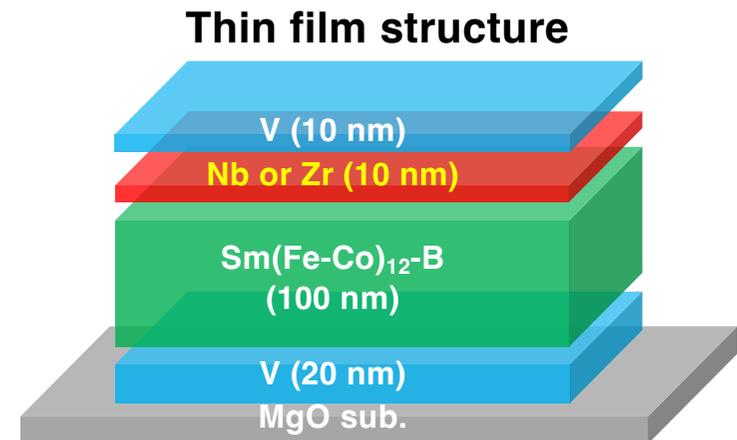
Experimental procedure

Sample preparation

- UHV magnetron sputtering system
- Substrate : MgO (100) single crystal
- Targets : Sm, Fe, $\text{Fe}_{50}\text{Co}_{50}$, $\text{Fe}_{80}\text{B}_{20}$, V
- Sub. temp. : $T_s = 400\text{ }^\circ\text{C}$
- Buffer layer : V (20 nm)
- Main layer : $\text{Sm}(\text{Fe-Co})_{12}\text{-B}$ (100 nm)
- Cap layer : Zr, Nb (10 nm)
- Ann. temp. : $T_a = 400 \sim 650\text{ }^\circ\text{C}$
- Ann. time : 1 hour
- Cover layer : V (10 nm) depo@R.T.

Characteristic measurement

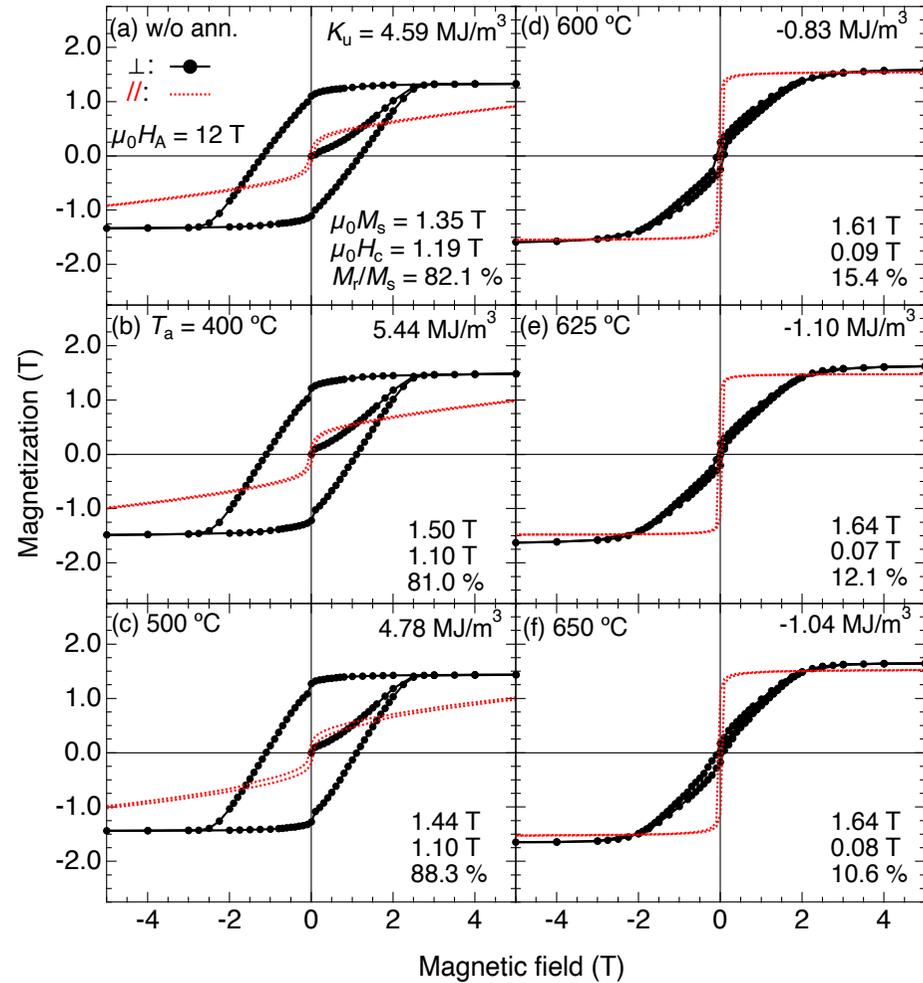
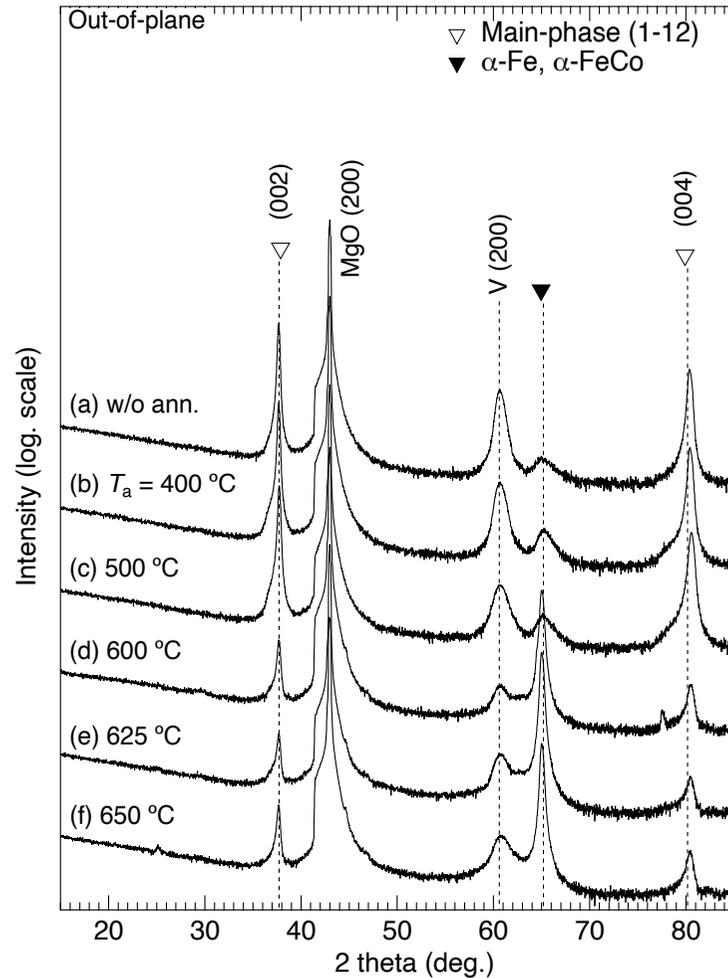
- Crystal structure : XRD
- Magnetic properties : SQUID



1) $\text{Sm}(\text{Fe-Co})_{12}\text{-B}$ thin films with different T_a

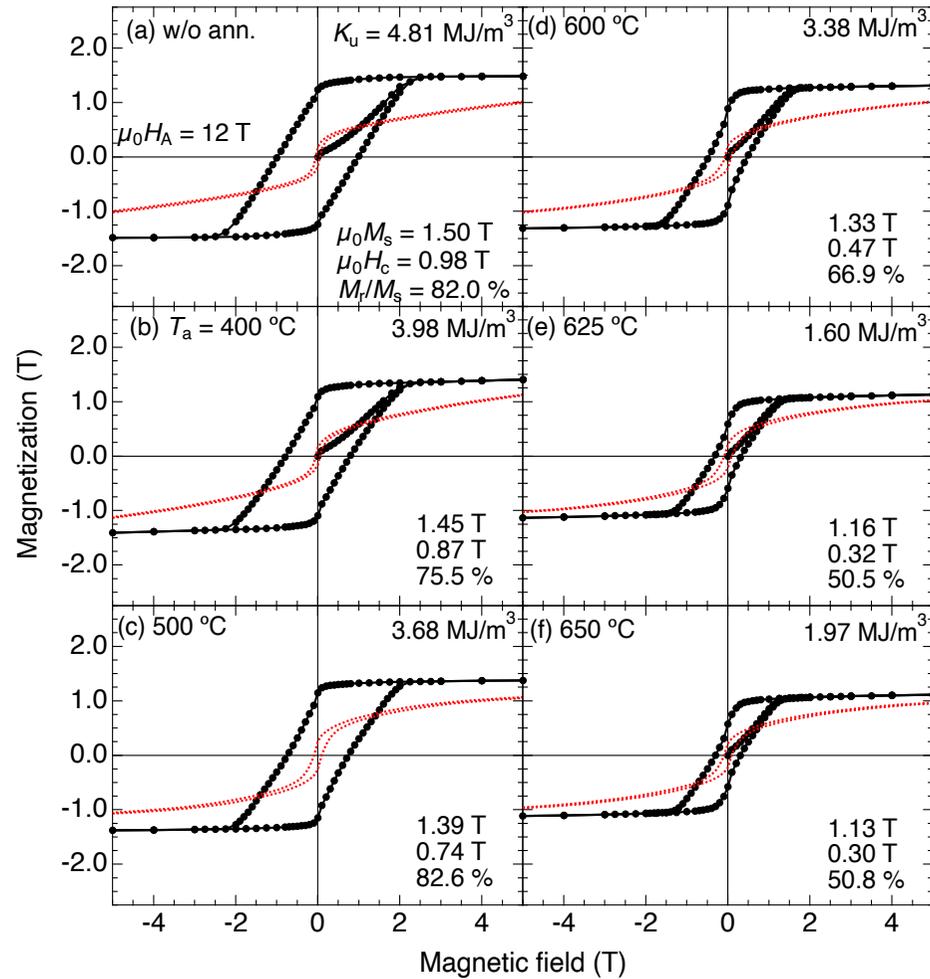
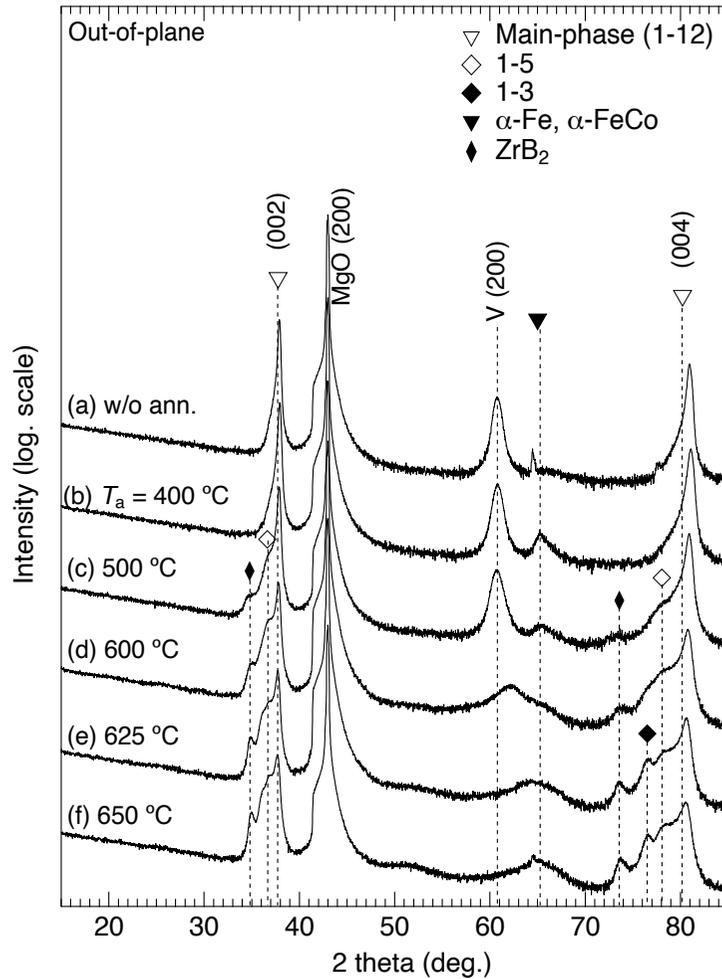
MgO sub./ V (20 nm)/ $\text{Sm}(\text{Fe-Co})_{12}\text{-B}$ (100 nm)/ V (10 nm)

$T_a = 400 \sim 600^\circ\text{C}$ (1 h)



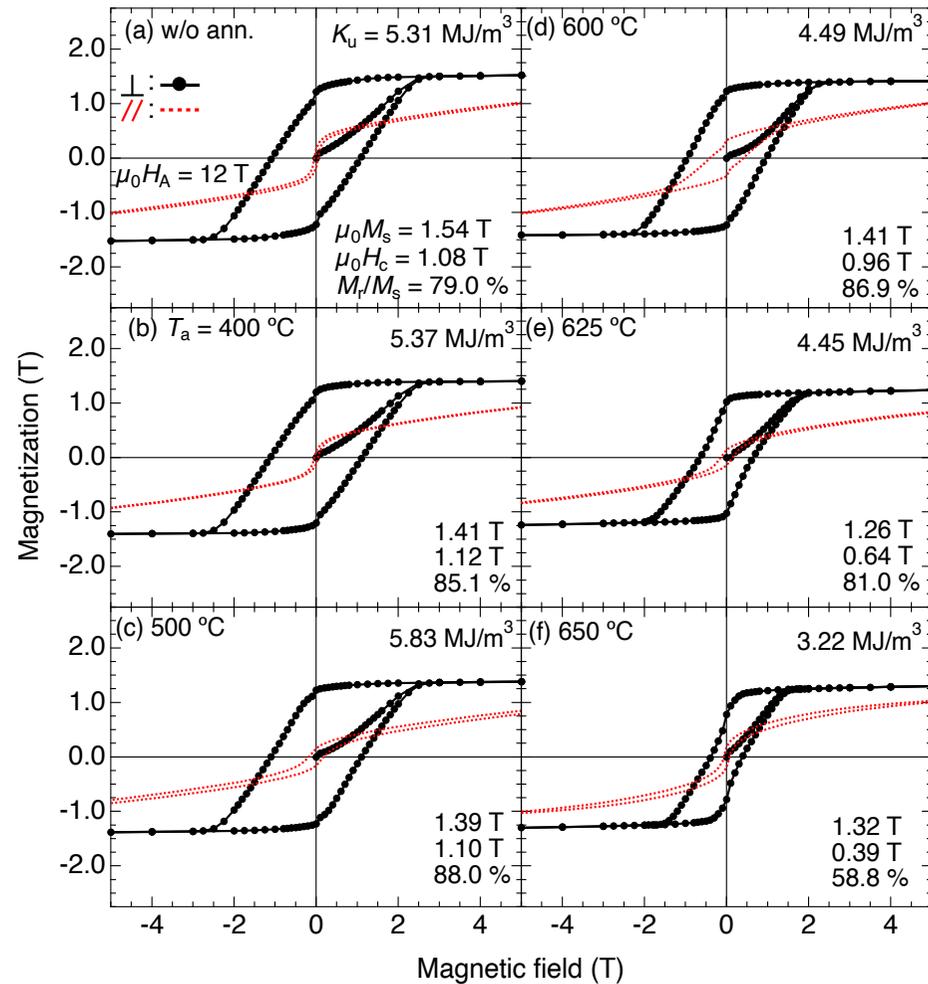
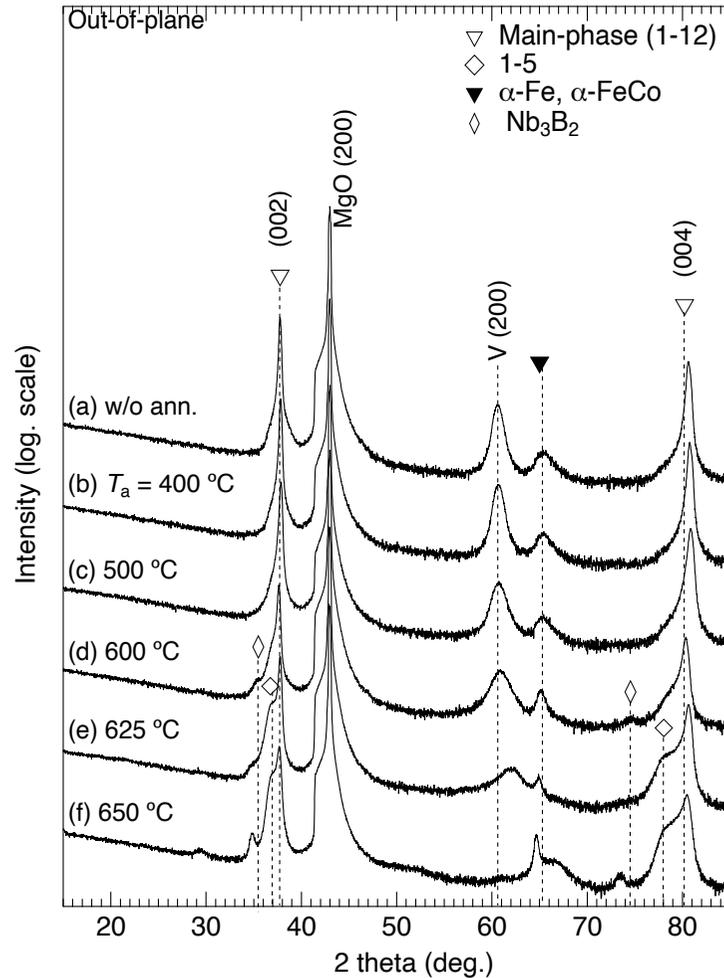
2) $\text{Sm}(\text{Fe-Co})_{12}\text{-B/Zr}$ thin films with different T_a

MgO sub./ V (20 nm)/ $\text{Sm}(\text{Fe-Co})_{12}\text{-B}$ (100 nm)/ **Zr (10 nm)/ V (10 nm) $T_a = 400 \sim 600^\circ\text{C}$ (1 h)**

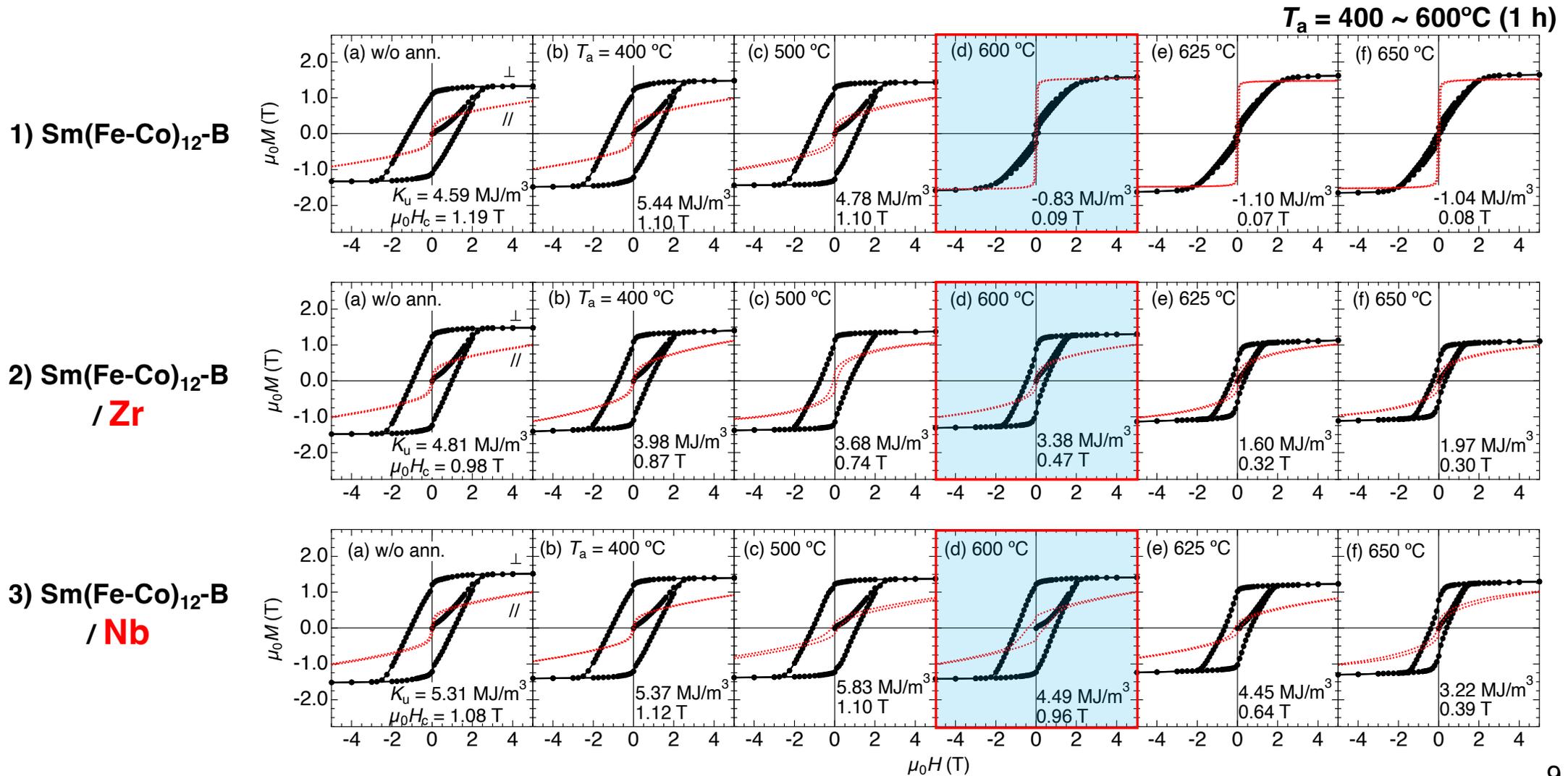


3) Sm(Fe-Co)₁₂-B/Nb thin films with different T_a

MgO sub./ V (20 nm)/ Sm(Fe-Co)₁₂-B (100 nm)/ Nb (10 nm)/ V (10 nm) $T_a = 400 \sim 600^\circ\text{C}$ (1 h)



M-H curves for Sm(Fe-Co)₁₂-B, Sm(Fe-Co)₁₂-B/Zr, and Sm(Fe-Co)₁₂-B/Nb thin films



Summary

In this study, in order to control the structure and improve the magnetic properties, stabilizing element such as Nb and Zr was selected as capping layer materials and post-annealed for $\text{Sm}(\text{Fe-Co})_{12}\text{-B}$ thin film.

1) $\text{Sm}(\text{Fe-Co})_{12}\text{-B}$

- Highly oriented 1-12 phase was obtained below 500°C , but it was decomposed above 600°C .
- High $\mu_0 H_c$ of 1.2 T was obtained in the deposited state but it decreased when T_a was increased to 600°C .

2) $\text{Sm}(\text{Fe-Co})_{12}\text{-B / Zr}$

- Decomposition of 1-12 phase was suppressed even at 600°C .
- High T_a resulted in the formation of ZrB_2 and 1-5 phases.
- $\mu_0 H_c$ of 0.47 T with perpendicular magnetic anisotropy was obtained at 600°C .

3) $\text{Sm}(\text{Fe-Co})_{12}\text{-B / Nb}$

- Decomposition of 1-12 phase was suppressed.
- Peaks from NbB and 1-5 phase were observed at high T_a .
- High $\mu_0 H_c$ of 0.96 T was obtained at 600°C .

Detailed microstructural analysis by APT and HRTEM is currently being performed.

Post annealed at $T_a = 600^\circ\text{C}$

