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TEM-Analysis of Sm(Co,Fe,Cu,Zr)_z magnets for high temperature applications

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Abstract

The influence of the chemical composition on the microstructure of high temperature Sm(Co,Fe,Cu,Zr)_z magnets is examined in this study. A completely developed cellular precipitation structure is the key factor for a high coercivity. Changing the chemical composition influences the microstructure as well as the microchemistry of the precipitations.

Keywords

Transmission electron microscopy, Microstructure, Rare earth – permanent magnets, Domain wall pinning

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Precipitation hardened $\text{Sm}(\text{Co},\text{Fe},\text{Cu},\text{Zr})_z$ permanent magnets are the most promising candidates for advanced power applications at high temperatures up to 500°C . There is a strong relationship between the microstructure and the magnetic properties [1,2]. For applications at high temperatures it is necessary to improve the domain wall pinning at the precipitations, which can be achieved via an optimized material composition. In this transmission electron microscopy (TEM) study the influence of the additives Fe, Cu and Zr on the microstructure is investigated. 7 different series of $\text{Sm}(\text{Co}_{\text{bal}}\text{Fe}_{0-0,14}\text{Cu}_{0,06-0,13}\text{Zr}_{0,02-0,04})_{6,8-9,5}$ are examined in order to analyze the microstructure-properties relation. There are differences in the cellular geometry as well as in the chemical composition of the several phases.

The production process and the heat treatment of the magnets as well as the magnetic data are described in another paper [3]. The TEM samples were prepared by mechanical grinding and afterwards ion milled with a GATAN Precision Ion Polishing System. The Observations were performed with a JEOL 200CX transmission electron microscope.

Transmission electron micrographs show a cellular precipitation structure which consists of a $\text{Sm}_2(\text{Co},\text{Fe})_{17}$ cell matrix, a $\text{Sm}(\text{Co},\text{Cu})_5$ cell boundary phase and a Zr-rich platelet phase perpendicular to the c-axis [4]. The cell boundaries act as pinning centers for the domain walls. Therefore a complete cellular structure is a necessary prerequisite for a high coercive field. The platelet phase is a necessary diffusion path for Cu and Fe during the heat treatment.

A higher Sm content results in a higher volume fraction of the $\text{Sm}(\text{Co},\text{Cu})_5$ cell boundary phase which may either lead to smaller cell dimensions or to thicker cell boundaries. Micromagnetic simulations show that a minimum thickness of 9 nm is necessary to achieve a high coercivity [5]. Fig.1 shows the cellular structure for the series $\text{Sm}(\text{Co}_{0,75}\text{Fe}_{0,14}\text{Cu}_{0,08}\text{Zr}_{0,04})_z$ with $z=8,7$ and $z=7,6$ where the cell size

decreases from 160 nm for $z=8,7$ (10,3 at.% Sm) to 60 nm for $z=7,6$ (11,6 at.% Sm).

All investigated series showed this correlation between cell size and Sm content independently of the chemical composition, but the absolute values are also connected to the heat treatment [6]. Samples with a higher Sm content tend towards an incompletely developed cellular precipitation structure. On the other hand a small cellular structure results in a lower temperature coefficient of coercivity β [7], which shows the need for an optimized heat treatment depending on the Sm content.

Even samples with no Fe at all show a perfectly developed microstructure which evidences that the Fe content does not necessarily show an influence on the microstructure. But the production process seems to be very sensitive on the Fe content. Fig.2 shows the differences in the microstructure of

$\text{Sm}(\text{Co}_{0,77}\text{Fe}_{0,07}\text{Cu}_{0,13}\text{Zr}_{0,03})_z$ with $z=7,6$ and $z=6,9$. The sample with $z=7,6$ exhibits a „typical“ cellular precipitation structure, whereas the microstructure of the sample with $z=6,9$ might be described as a clustering of very small single cells combined with some very large cells. This is consistent with the observations by Liu [8] and Tang [9] that low Fe content together with high Sm content influences or even destroys the cellular character of the precipitation structure, but that the microstructure of samples with low Sm is not influenced. Obviously further studies on the heat treatment procedure for magnets with low Fe content are necessary.

Electron nanoprobe analysis showed that Cu mainly segregates in the cell boundary phase [10]. A higher Cu content in the $\text{Sm}(\text{Co}_{1-x}\text{Cu}_x)_5$ cell boundary phase decreases the magnetocrystalline anisotropy [11]. As the pinning field is proportional to the difference of the magnetocrystalline anisotropy between the 2:17 cell matrix phase and the 1:5 cell boundary phase, a high Cu content increases the coercivity [12]. But Cu has also an influence on the platelet phase. A high Cu content favors the occurrence of microtwinning instead of the formation of platelets. Fig.3 shows a

comparison between $\text{Sm}(\text{Co}_{\text{bal}}\text{Fe}_{0,14}\text{Cu}_x\text{Zr}_{0,02})_8$ with $x=0,076$ and $x=0,127$. The higher Cu content results in a very high microtwin density of 180 microtwins/ μm compared to the platelet structure in the sample with moderate Cu content with a platelet density of $22 \mu\text{m}^{-1}$. The platelet density changes rapidly throughout the sample but becomes a constant at a length scale of about 2 microns. High coercive magnets tend towards higher platelet densities up to $50 \mu\text{m}^{-1}$, but a direct correlation between platelet density and coercivity could not be observed. Zr stabilizes the hexagonal $\text{Sm}_2\text{Co}_{17}$ platelet phase even in samples with high Cu content. Independently of the Cu content a high Zr content enables thicker platelets (Fig.4).

Electron microprobe analysis [13] showed that only approximately 3 at.% Zr are soluble in the 1:5/2:17 phases. The excess Zr forms Zr-rich phases like $\text{Zr}_6\text{Co}_{23}$. The size of these grains shows a distribution from about 100nm up to several microns.

In conclusion the influence of Fe, Cu and Zr on the microstructure is always dependent on the Sm content. A variation of the Sm content results in a variation of the volume fractions of the several phases and therefore changes the microchemistry. The formation of the platelet phase is influenced by both, Cu and Zr. The formation of the cellular precipitation structure is sensitive on the Fe content and requires an optimized heat treatment.

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Figure captions

Fig.1 TEM micrographs of $\text{Sm}(\text{Co}_{0,75}\text{Fe}_{0,14}\text{Cu}_{0,08}\text{Zr}_{0,04})_z$ with $z=8,7$ (a) and $z=7,6$ (b).

Fig.2 TEM micrographs of $\text{Sm}(\text{Co}_{0,77}\text{Fe}_{0,07}\text{Cu}_{0,13}\text{Zr}_{0,03})_z$ with $z=7,6$ (a) and $z=6,9$ (b).

Fig.3 TEM micrographs of $\text{Sm}(\text{Co}_{\text{bal}}\text{Fe}_{0,14}\text{Cu}_x\text{Zr}_{0,02})_8$ with $x=0,076$ (a) and $x=0,127$ (b).

Fig.4 TEM micrographs showing the platelet phase of $\text{Sm}(\text{Co}_{0,75}\text{Fe}_{0,14}\text{Cu}_{0,08}\text{Zr}_{0,04})_{7,6}$ (a) and of $\text{Sm}(\text{Co}_{0,70}\text{Fe}_{0,14}\text{Cu}_{0,13}\text{Zr}_{0,04})_{9,3}$.

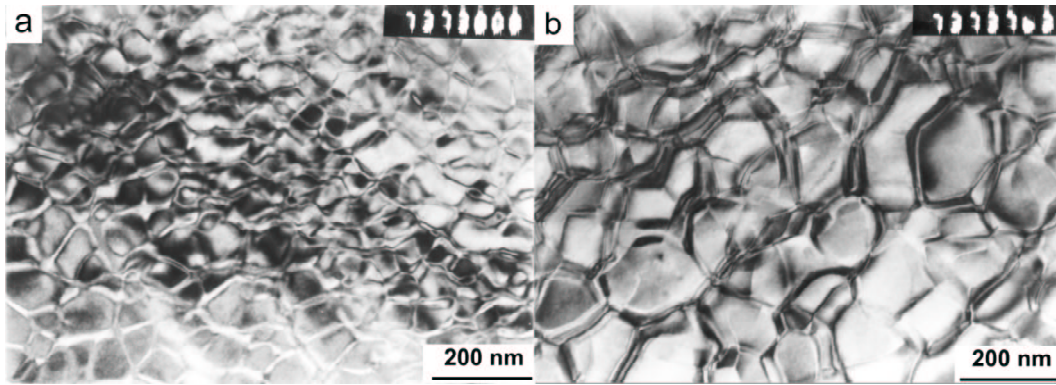


Fig.1 T. Matthias, J034

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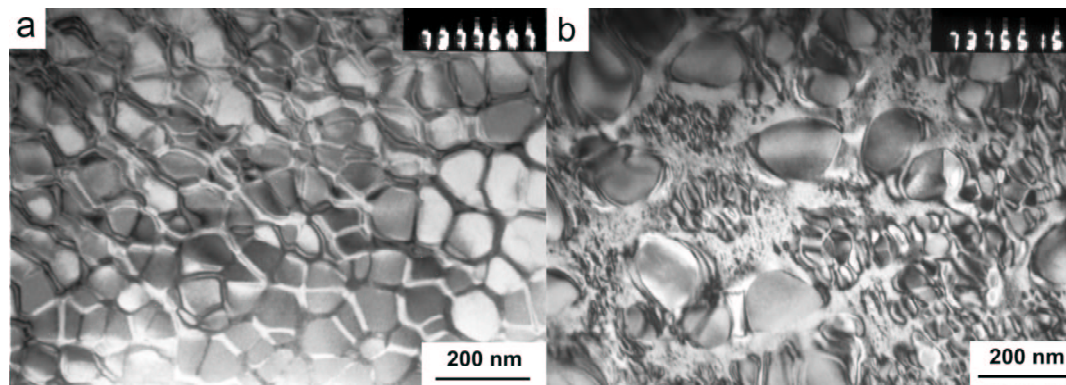


Fig.2 T. Matthias, J034

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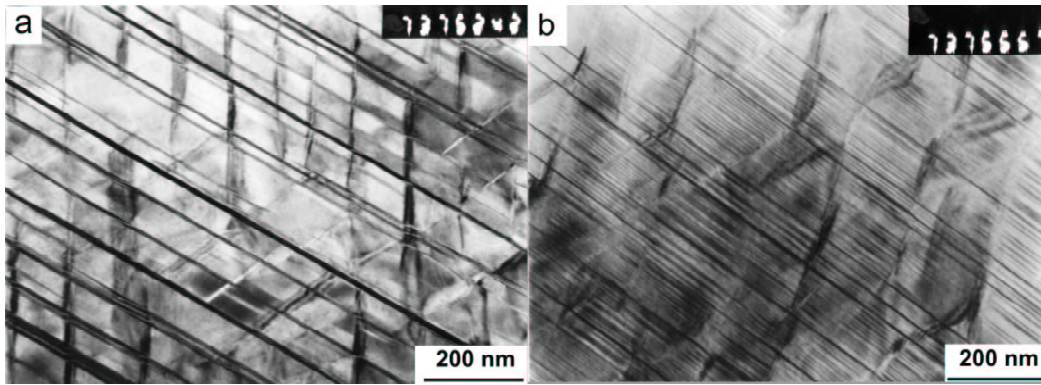


Fig.3 T. Matthias, J034

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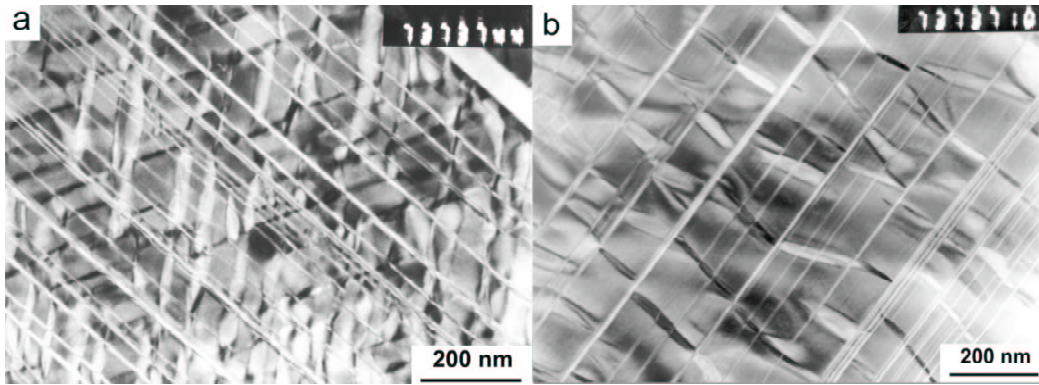


Fig.4 T. Matthias, J034

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