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Journal of Alloys and Compounds 429 (2007) 343–347

Journal of ALLOYS AND COMPOUNDS

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On the nature of the disordered microstructure in $Sm(Co,Cu)_5$ alloys with increasing Cu content

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Received 22 February 2006; received in revised form 20 April 2006; accepted 22 April 2006
Available online 30 May 2006

Abstract

X-ray diffraction and transmission electron microscopy (TEM) experiments were performed in heat-treated $Sm(Co,Cu)_5$ samples with different Co/Cu content. The major phase observed in the diffraction patterns exhibits a hexagonal $CaCu_5$ type structure in all the studied compositions. The behavior of the diffraction profiles, as function of the Cu content, is believed to be due to an inhomogeneous distribution of Co and Cu through out the samples. Heavily planar faulted regions have been observed by means of TEM and are also associated with Sm and Co rich phases within the matrix. A $CaCu_5$ type structure with local Sm rich regions seems to be responsible for the observed magnetic behavior.

Keywords: Sm–Co alloys; X-ray diffraction; Electron microscopy; Structural disorder

1. Introduction

$SmCo_5$ is a well-known compound which exhibits extremely high magnetocrystalline anisotropy [1,2]. Together with Sm_2Co_{17} they belong to a binary family of compounds well suited for permanent magnets. Actually the crystal structure of both compound are closely related, and Sm_2Co_{17} can be considered resulting from the ordered substitution of Sm atoms by a pair of Co atoms which are usually termed "dumbbells" [3]. When the substitution of rare earth atoms by the Co atoms is not complete, a disordered structure has been reported with the generic name of $SmCo_7$. On the other side of the phase diagram the phase Sm_2Co_7 appears, this compound also shows high magnetocrystalline anisotropy, and it is also closely related to the $SmCo_5$ structure. For the binary Sm–Cu alloys the $SmCu_5$ can also be found suggesting a complete solubility of Cu in the $SmCo_5$ structure. In previous studies it has been shown that the addition of Cu to the ferromagnetic compound $SmCo_5$ results in a permanent magnet with high values of coercive field [4–7]. The achievement of such high coercivity occurs in spite of the lowering in the magnetocrystalline anisotropy. The coercivity depends strongly on the thermal treatment of the sample and with Cu content, a transition of demagnetization mechanism is observed from a nucleation type magnet to a pinning type magnet [8,9].

Surprisingly, the microstructure of the Cu substituted $Sm(Co–Cu)_5$ does not show a complete solubility of Cu in the $SmCo_5$ matrix even when subjected to a 4 week heat treatment. Observed by optical, Kerr and electron microscopy, the microstructure shows the occurrence of heavy disorder in the samples and strong evidence of planar faulting was observed by transmission electron microscopy (TEM) [10–12]. The same disorder was observed by optical microscopy indicating that the observed features were not result of sample preparation in the TEM samples.

There is still discussion on the nature of the disorder in Cu substituted $SmCo_5$ compounds [13–16]. While it is believed that Cu enhances the Sm solubility in the alloy driving the compound towards the Sm_2Co_7 phase, there is also indications that disordered $SmCo_7$ phase can also be present in the heat treated sample.

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doi:10.1016/j.jallcom.2006.04.044

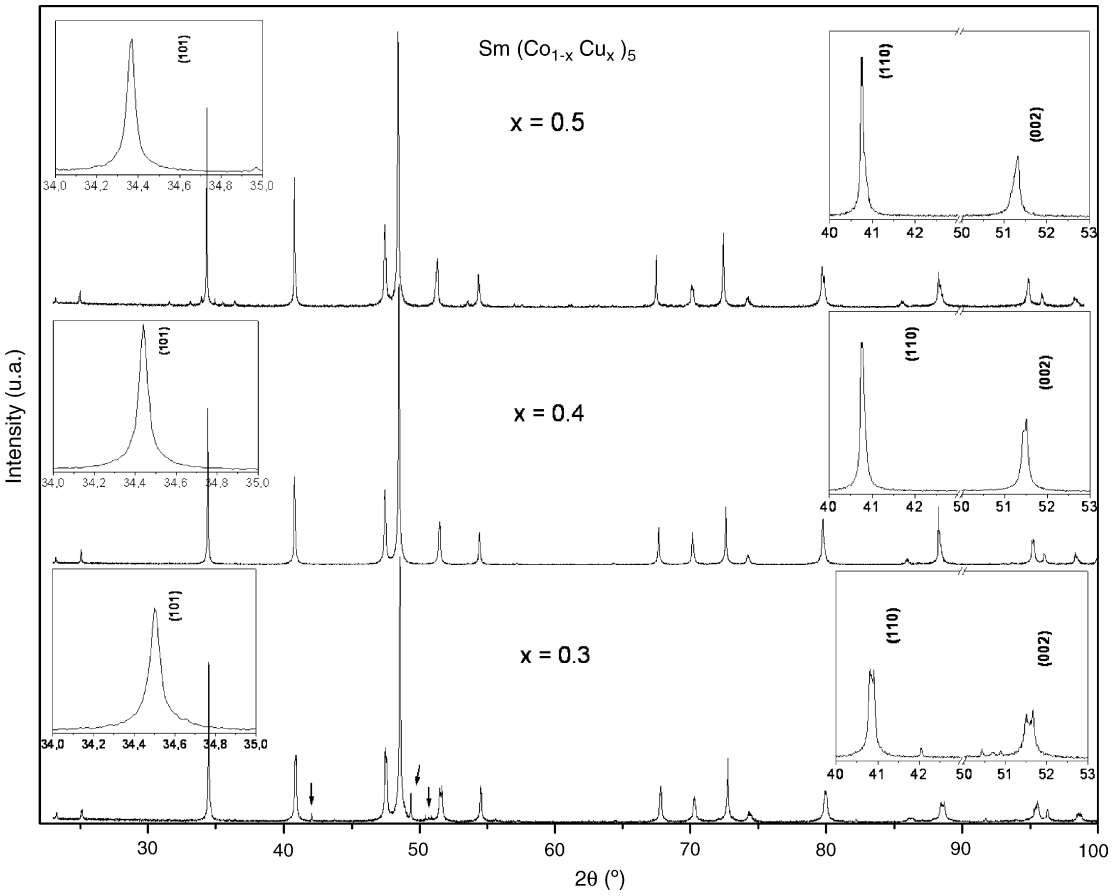

Fig. 1. X ray diffraction pattern of the studied compositions with a $CaCu_5$ type structure as a major phase. The insets show the behavior of the peak profiles as function of Cu content.

We report on the microstructural and structural study of $Sm(Co,Cu)_5$ samples with increasing Cu content. The idea is to study the kind of disorders occurring in the alloys with Cu content. Studies were carried out by high resolution X-ray diffraction and TEM. X-ray diffraction will give information on the crystal structure and the microstructure of whole of the material while TEM, can shed light on the local variations of the microstructure.

2. Experimental

Samples of $Sm(Co_{1-x}Cu_x)_5$ alloys with $x = 0.3$, 0.4 and 0.5 were prepared by induction melting using raw material with purity of at least 99.9 wt.%. The as cast ingots were embedded in a Ta foil and placed in a quartz tube, which was evacuated prior to filling with purified Ar gas and then annealed at 1000 °C for 2 weeks. In order to compensate for Sm loss at high temperature, a small piece of metallic Sm was placed in the quartz tube.

During the cooling process, the annealed samples were subsequently water—quenched to room temperature. Finally, samples were reduced to powder in order to perform X-ray diffraction experiments.

X-ray experiments were conducted on beamline XRD at the LNLS, Synchrotron facility in Campinas, Brazil. The Bragg–Brentano optical system consisted of a bending magnet light source, a double—crystal Si (1 1 1) monochromator and a Ge (1 1 1) crystal analyzer, the specimen was mounted in a 10 mm diameter rotating sample holder, the radiation energy was 7098.6 eV which corresponds to a wavelength of 1.7466 Å. The powder diffraction data was collected at room temperature, with a step of 0.01° and a time count of 1 s.

From the obtained alloys, samples were cut as 3 mm diameter disks. The disks were ground and polished to electron transparency in an Argon ion mill at 4 kV. The samples were examined in a 200 kV TEM.

3. Results

Fig. 1 shows the high resolution X-ray diffraction patterns of the studied compositions. The diffraction pattern could be indexed assuming a hexagonal unit cell with space group *P*6/*mmm* corresponding to the $CaCu_5$-type structure. For the $x = 0.3$ composition, additional peaks (marked with arrows) were found. These additional peaks could be indexed as a rhombohedral crystal structure with the Th_2Zn_{17}-type structure (Sm_2Co_{17} phase) and space group *R*-3*m*. The Sm_2Co_{17} phase disappears with increasing Cu content and its reflections are absent for $x = 0.4$, 0.5. Some additional peaks that appears for the composition $x = 0.5$ were identified as SmO_2, phase that arises due to the oxidation of the sample.

The insets in Fig. 1 show the peak profile behavior for reflections (1 1 0) and (0 0 2) as function of the Cu content and they were chosen as representative of all the other maxima of the diffraction pattern. In the case of $x = 0.3$ the diffraction maxima exhibit broadened asymmetric splitted profiles. The splitting is reduced with increasing Cu content but even for $x = 0.5$ asymmetric broadening is still observed. Reflections like (h 0 h), i.e.