Magnetic properties of mechanically alloyed Co–Ti powder

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Abstract

An amorphous phase containing traces of non-transformed Co and Ti powders was obtained by mechanical alloying nominal compositions of $Co_{67}Ti_{33}$ and $Co_{50}Ti_{50}$ in a high-energy ball-mill. These alloys were prepared from elemental powders of Co and Ti. The heat treatment of $Co_{67}Ti_{33}$ at 573, 873 and 1173K crystallized nanoparticles of Co_2Ti and Co_3Ti compounds, while the same treatments conducted on $Co_{50}Ti_{50}$ resulted in the formation of Co_2Ti and CoTi nanoparticles. The saturation magnetizations reached a maximum value in the amorphous state and they decreased when the temperatures of the heat treatment rose. Demagnetizing interparticle interaction effects were estimated through hysteresis loops and initial magnetization curves using the Fourier technique.

Keywords: Magnetic alloys; Mechanical alloying; Magnetic properties.

Mechanical alloying of the Co–Ti system shows the formation of an amorphous-like phase afte long-term milling and the crystallization of Co–Ti compounds after hot consolidation operations. On heating, the mechanically alloyed Co–Ti powders undergo a rather complex crystallization sequence. In addition to a nucleation kinetics effect, Fe and Cr contaminants are the probable factors which produce a stabilization effect to let Co_2Ti (hex) and Co_3Ti phases precipitate from the amorphous phase ($Co_{67}Ti_{33}$ composition), rather than the thermodynamically predicted Co_2Ti (cubic) and CoTi [1]. In this work the microstructural characterization of this amorphous phase and its phase transformation upon heating is further analyzed for two different Co-concentrations as an attempt to simplify and decipher its complex crystallization behavior.

The compositions $Co_{67}T_{33}$ and $Co_{50}Ti_{50}$ correspond to two stoichiometric Co:Ti extensions of the equilibrium phase diagram. They were prepared from elemental Co and Ti powders (99.9% and particle sizes of 2 and 40 mm, respectively) and ball milled for 4 h under argon atmosphere in a high-energy SPEX ball milling device. Stainless steel balls of ϕ 10mm were used as milling media and a ball-to-powder ratio was set 10:1.

$Co_{66.4}Fe_{0.6}Ti_{33}$ and $Co_{48.6}Fe_{1.0}Ti_{50.4}$ were found as the final milling products for the initial compositions $Co_{67}Ti_{33}$ and $Co_{50}Ti_{50}$, respectively. In both cases the Co and Ti contents are very close to the nominal composition, it is evident that the different final composition may be due to mechanical drag, and/or because the elemental powder were embedded in the milling media. Iron traces were mostly incorporated into the as-milled powder mixtures due to erosion of the milling media. In contrast to previous work [1], the Fe concentration seems not to considerably distort the final composition. After milling, diffraction peaks corresponding to elemental Co and Ti were observed in combination with an amorphous-like pattern (Fig. 1). The Fe contaminant seems not to have any influence on the Co–Ti mixtures amorphization sequence [2].

Differential scanning calorimetric analysis revealed very similar curves and overlapping of several exothermic peaks on heating the $Co_{66.4}$- $Fe_{0.6}Ti_{33\ a}$nd $Co_{48.6}Fe_{1.0}Ti_{50.4}$ samples. These exothermic events are due to crystallization of the amorphous phase for producing intermetallic compounds. The peaks were generally found between 573 and 873 K. To further understand the intermetallic formation, a fewgrams of milled specimens were separately heated at 573, 873 and 1173K and characterized by XRD (Fig. 1).

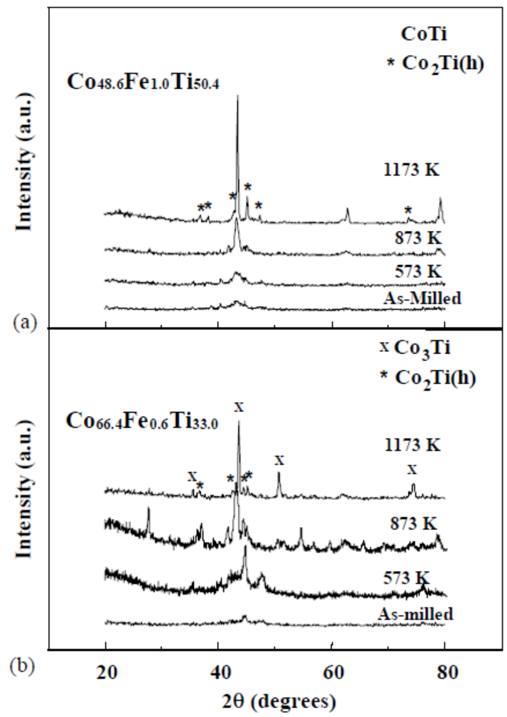

Fig. 1. XRD patterns of (a) $Co_{48.6}Fe_{1.0}Ti_{50.4}$ and (b) $Co_{66.4}Fe_{0.6}Ti_{33}$ after being heated at different temperatures. Intermetallic compounds are formed upon crystallization.

Table 1
XRD generated data of mechanically alloyed mixtures heated up to 1173 K

Mixture	Phases	a_0 (nm)	wt%
$Co_{48.6}Fe_{1.0}Ti_{50.4}$	CoTi	$a = 0.296$	85.55
$Co_{48.6}Fe_{1.0}Ti_{50.4}$	$Co_2Ti_{(hex)}$	$a = 0.472$ $c = 1.544$	14.45
$Co_{66.4}Fe_{0.6}Ti_{33}$	Co_3Ti	$a = 0.360$	86.55
$Co_{66.4}Fe_{0.6}Ti_{33}$	$Co_2Ti_{(hex)}$	$a = 0.473$ $c = 1.542$	13.45

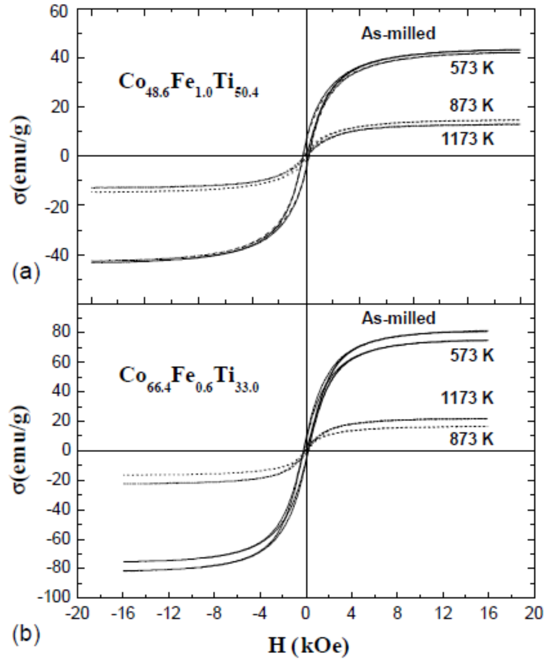

Fig. 2. Hysteresis loops of (a) $Co_{48.6}Fe_{1.0}Ti_{50.4}$ and (b) $Co_{66.4}$-$Fe_{0.6}Ti_{33}$ after being heated at different temperatures.

For the composition $Co_{48.6}Fe_{1.0}Ti_{50.4}$ the 573K pattern looks like the one in the as-milled condition. At 873 K, a cubic (CsCl-type) intermetallic compound CoTi is growing from the amorphous matrix together with the incipient presence of the Co_2Ti compound. At 1173K the CoTi and Co_2Ti phases grew up and showlarger and sharper diffraction peaks. The composition $Co_{66.4}Fe_{0.6}Ti_{33}$ (Fig. 1b) shows the crystallization of the Co_2Ti and Co_3Ti compounds with a similar evolution. Table 1 summarize the phases identified after heat treatment (DSC), their volume fraction and lattice parameter. The hysteresis loops (Fig. 2) shows that the saturation magnetization reaches a maximum value in the amorphous state and decreases when the temperatures of the heat treatment rise.

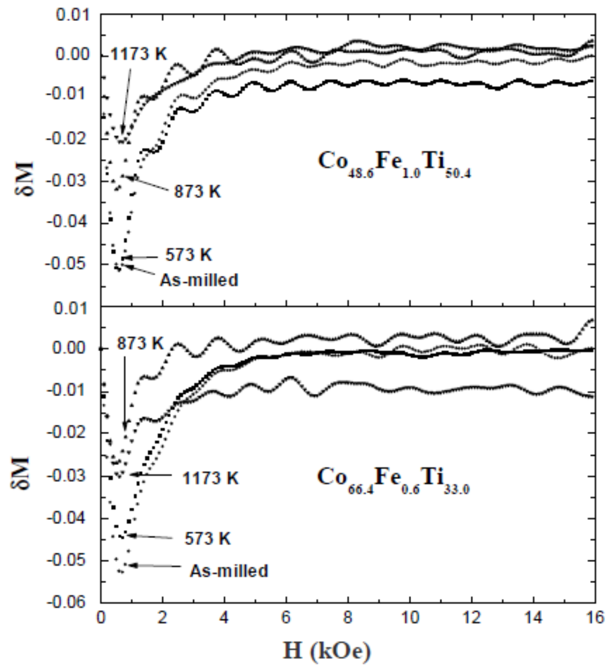

Fig. 3. δM vs. H of (a) $Co_{48.6}Fe_{1.0}Ti_{50.4}$ and (b) $Co_{66.4}Fe_{0.6}Ti_{33}$ after being heated at different temperatures obtained by using the Fourier technique.

For both compositions the hysteresis loops are similar for the as-milled and the 573 K, the same for the 873 and 1173K samples. The effects of interaction in particulate media are of considerable interest because they can influence substantially the magnetic characteristics of the media [3,4]. These effects are a complex phenomenon, which is far from being well understood. Usually, remanence measurements are used for estimating the interactions. However, the hysteresis loops contain valuable information concerning the magnetic interactions, which can be extracted by using Fourier techniques. Fig. 3 shows the δM vs. H curves obtained for both compositions. Demagnetizing interparticle interaction effects were estimated through hysteresis loops and initial magnetization curves using the Fourier technique.

For the composition $Co_{48.6}Fe_{1.0}Ti_{50.4}$ the particle interactions are stronger and similar for the asmilled and the 573K samples and decreases for 873 and 1173 K. For the composition $Co_{66.4}Fe_{0.6}$- Ti_{33} the particle interactions are stronger for the as-milled sample and decreases with the heat treatment. At 873 and 1173K the interaction are similar (Table 1).

Mechanically alloyed Co–Ti powders consist of nanocrystallites dispersed in an amorphous-like matrix. During heat treatment, the amorphouslike phase transform into a mixture of intermetallic compounds. For both compositions the saturation magnetization reaches a maximum value in the amorphous state and decreases when the temperatures of the heat treatment rise. Demagnetizing interparticle interaction effects were estimated stronger for the as-milled state and decrease with the treatment temperature.

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