XRD Studies of Co-Fe-Ti Nanocrystalline Intermetallics

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\textbf{Keywords:} Co-Ti Intermetallics, Mechanical Alloying, Nanocrystalline Materials, Plasma Sintering

\textbf{Abstract.} Cobalt-rich Co-Fe-Ti alloys, produced by spark plasma sintering of mechanically alloyed powders, possess a microstructure consisting of an intimate mixture of nanocrystalline intermetallic phases. However, some of these phases, particularly the Co\textsubscript{2}Ti phases, have not been thoroughly characterized before, and the presence of iron opens several possibilities regarding the location of iron atoms in the crystalline lattices. In the present study, x-ray diffraction methods have been applied to characterize such microstructures, by evaluating the crystallite size, microstrain, volume fraction of phases and lattice parameters of the materials.

\textbf{Introduction}

In the past, we have reported the production of Co-Fe-Ti alloyed powders obtained by mechanical alloying of Co-Ti elemental powder mixtures which, during the mechanical alloying process itself, picked up substantial amounts of iron from the milling media [1-5]. For the most part, the structures found in the mechanically alloyed powders consist of a dispersion of nanocrystalline phases within a pseudo-amorphous matrix (i.e., a truly amorphous phase together with crystallites having sizes smaller than about 3 nm). In bulk alloys containing more than 60 at\% cobalt and produced by spark plasma sintering of the mechanically alloyed powders, these structures essentially turn into two-phases mixtures involving the intermetallic phases (Co,Fe)\textsubscript{2}Ti and (Co,Fe)\textsubscript{3}Ti. These alloys have been observed to maintain a nanocrystalline nature even after being heated to temperatures as high as 1000 °C [1-5].

The structures of the binary intermetallic compounds Co\textsubscript{2}Ti and Co\textsubscript{3}Ti have not been completely clarified in the open literature. Particularly, we have found some discrepancies in our analysis of x-ray diffraction data from the Co\textsubscript{2}Ti hexagonal phase [3]. Therefore, we have performed a more detailed investigation of the diffraction data from our Co-rich alloys with the intention to clarify the information obtained from these materials and the corresponding intermetallic phases.

\textbf{Experimental}

Elemental powders of Co and Ti (99.9\% purity), were used as starting materials in the mechanical alloying experiments. The initial composition (atomic percent) of the powder mixtures was Co\textsubscript{25}Ti\textsubscript{25}. Additional results obtained from samples of nominal compositions Co\textsubscript{0.7}Ti\textsubscript{1.33} and Co\textsubscript{0.9}Ti\textsubscript{1.0} were also used in our characterization, as well as data from a specimen of nominal composition Co\textsubscript{25}Ti\textsubscript{25} produced from a different batch of mechanically alloyed powders. The mechanical alloying experiments have been described in detail elsewhere [1,2].

The mechanically alloyed powders were hot consolidated by spark plasma sintering at temperatures of 800, 900, 1000 and 1100 °C, mostly under an uniaxial pressure of 50-60 MPa. The compacts so produced were in the shape of discs 25 mm in diameter and 2.5 to 3.0 mm thick.

The consolidated compacts were characterized by x-ray diffraction (XRD), scanning electron microscopy (SEM) and by energy-dispersive spectrometry (EDS). XRD was performed using Cu Kα and Cr Kα radiations, so that differences in the intensities of the peaks due to the fluorescence phenomenon could be used to improve the determination of atomic positions. The program Fullprof