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Mechanically induced instability in Fe_2Ti and mechanical alloying of Fe and Ti

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Mechanical alloying (MA) of Fe and Ti elemental powder mixtures has been reported in studies mainly related to the preparation of nanocrystalline FeTi intermetallic compound [1–8]. In all these studies amorphous and/or nanocrystalline phases have been reported as end products of the milling process. The atomic composition 2Fe:1Ti has received less attention. For this composition the formation of amorphous phase on milling has been reported [2]. The amorphous phase was characterized by a broad X-ray diffraction peak and its crystallization was observed on heating above 800 K [2].

A more recent paper on mechanical milling (MM) of Fe_2Ti reports the occurrence of certain structural disorder in the C14 hexagonal structure of this Laves phase but without the formation of an amorphous material [9]. The structural disorder is related to the occurrence, under milling, of planar defects such as stacking faults. According to X-ray diffraction (XRD) annealing at 873 K of this disordered phase restores the crystal structure of Fe_2Ti. However, from the Mössbauer spectrum of the annealed of MM Fe_2Ti sample about 5% of bcc iron is reported [9], which could be interpreted as an incipient decomposition process of Fe_2Ti due to the milling process. In that case, on prolonged milling a higher amount of bcc iron would be formed. In this contribution we report the resulting intermediate and end products of 2Fe:1Ti elemental mixtures and Fe_2Ti powder samples when they were submitted to prolonged milling. In both cases in the end products a solid solution of Ti in α-Fe, α-Fe(Ti) (bcc), was detected.

The intermetallic compound Fe_2Ti was prepared by melting the appropriate mixture of elemental Fe (99.9% nominal purity) and Ti (99.9%) coarse powders in an electric arc-furnace under an argon atmosphere. After melting, the alloy was sealed in a quartz tube filled with argon and then annealed for 48 h at a temperature of 1173 K to aid homogeneity and crystallinity. Its elemental and phase composition was determined using energy-dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD), respectively.

Prior to milling, the Fe_2Ti ingot was cut into small pieces using a diamond saw. The powder resulting from the cutting process was collected and used as representative of the starting Fe_2Ti sample (without milling).

For the MA of 2Fe:1Ti elemental mixtures, iron and titanium powders of 3 and 250 μm of particle size, respectively and with a nominal purity of 99.9 wt% were used. The milling process of both, the intermetallic Fe_2Ti and the mixtures of 2:1Ti mixed powders was carried out using a high energy ball mill (Spex 8000D) with balls and vials made of hardened steel. The powders were loaded and sealed into the vials inside a glove box filled with argon (99.99% of purity). Removal of milled samples at different milling times was also performed under argon atmosphere. The weight ratio of balls to powders varied from 10:1 to 5:1 in our experiments.

The ball-milled powders were characterized by X-ray diffraction (XRD) and Mössbauer techniques. XRD powder patterns were recorded using a Siemens D-500 diffractometer with monochromated Cu K_α radiation. Mössbauer spectra were obtained at room temperature using a ^{57}Co in Rh source and a constant acceleration spectrometer (from Wissel) operated in the transmission mode. All Mössbauer spectra were fitted with distributions of hyperfine magnetic field (B_H) and quadrupole splitting (Δ) [10]. In the fitting of Mössbauer spectra with a distribution of B_H, a linear correlation between the isomer (δ) and quadrupole splitting (Δ), $\delta = a + b\Delta$, was assumed. This means that the obtained value of δ can be used to follow the trend in the electronic interactions of the involved metals (Ti and Fe) but not as an exact measure of these interactions. Some spectra were also fitted with an iterative least-squares minimization algorithm using pseudo-Lorentzian line shapes. Isomer shift values are reported relative to metallic iron (α-Fe).

According to its composition and crystal structure Fe_2Ti belongs to the Laves phase C14 (hexagonal $MgZn_2$ type cell). In this structure there are eight iron atoms per unit cell located in two different structural sites, 2(a) and 6(f). This compound has an antiferromagnetic behavior with a layered spin structure where the magnetic moments on Fe(6f) are antiparallel along the c axis while Fe(2a) remains non-magnetic [11]. The Mössbauer spectra of Fe_2Ti at room temperature can be interpreted as a single quadrupole doublet with $\delta = -0.34$ mm/s (relative to α-Fe) and $\Delta = 0.49$ mm/s

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