Thermal stability of ball milled Fe-Al alloy

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Abstract

The ball milling method is widely spread in between solid-state technique of preparation micro-/nanocrystalline materials. It is suitable for mechanically alloying of various compounds by changing of parameter of preparation. The knowledge of phase composition of prepared material is essential for determination of magnetic, kinetic etc. behavior. The study of phase composition by standard method e.g. X-ray powder diffraction or Mössbauer spectroscopy is limited by resolution of method or by time of measurement.

High temperature measurement helps X-ray powder diffraction was applied for determination of the thermal stability of ball milled Fe-Al samples doped by Mo as well as for nucleation and growth of new phases. The study of magnetic properties in an iron-aluminum system [1] shows the comparable coercivity and saturation magnetization of mechanically alloyed and bulk materials. Analysis of heat treated samples milled for 960, 1920 and 3840 minutes exhibited phase the decomposition the prepared samples to the various phases (precursors, intermetallic's etc.) in dependence of milling time. The "ideal" sample remains stable even at high temperature.

Introduction

FeAl based alloys are interesting materials for their reasonable low cost, low density, high temperature corrosion resistance, and good intermediate-temperature mechanical properties [1]. The some of these systems show interesting magnetic properties. The most of these alloys are compound of Fe-Al solid solution with bcc structure and variety of intermetallic phases such as, e.g. Fe_3Al, FeAl, $Fe_{13}Al$.

The mechanical milling is well-established method of production of various materials, as nanocrystalline alloys, amorphous compounds, quasicrystals, etc. [2] and its helps us to dope Fe-Al matrix by small amount of Mo. We studied the thermal stability of this systems and molybdenum influence on its magnetic behavior.

Experimental

The sample was mechanical milled from high purity Fe (99.99%; µ10 m), Al (99.99%, 10 µm), and Mo (99,99%) powders. The high energetic milling was done using a Fritsch planetary ball mill Pulverisette 7 premium. To avoid sample heating the regime was chosen in such a way that 60 min of milling were followed by 60 min of pause (1 cycle). Powder samples were removed at specific times for the elemental and microstructural analysis. The nominal composition was chosen $Fe_{75}Al_{23,5}Mo_{1,5}$.

An X'PertPro diffractometer with Co Kα radiation ($\lambda = 0.17902$ nm) was used to study the structural changes occurring in the mixture in various stages during milling. The high temperature measurement was carried out by means HTK–1600 heating chamber in range 30-1000°C in vacuum (~10^{-2} Pa). Detailed analysis of powder patterns was realized using the Rietveld structure refinement method and the ICSD database of inorganic and related structures.

The measurements of temperature dependences of magnetic moments (thermomagnetic curves) and hysteresis loops were carried out by the vibrating sample magnetometer in the temperature range 30-800°C in vacuum (~10^{-1} Pa).

Results

The time of milling has the significant influence on the structure of alloyed material. The samples milled for short time contained all of tree initial metals (Fe, Al and Mo). The heat treatment caused incorporation of some compounds e.g. Mo and decomposition of Fe-Al solid solution to Fe_3Al and pure α-Fe (Fig. 1). The solid solution Fe-Al with trace of Mo is present in the sample milled for 1920 minutes. The residuum of Mo was dissolved during the heat treatment in Fe-Al and this phase remains stable even at 1000°C (Fig. 2). By means the prolongation of the milling time the amount of residual Mo did not decreased, but amount started growing again (Fig. 3) and the decomposition of solid solution to Fe_3Al and α-Fe is visible in the figure.

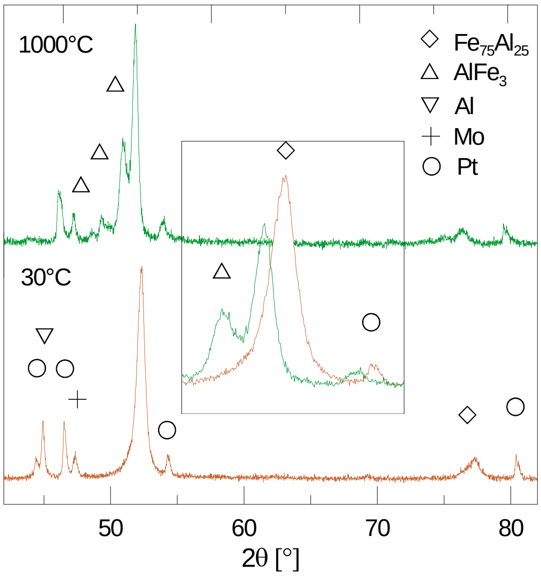

Figure 1. X-ray pattern milled (960 min) and annealed sample.

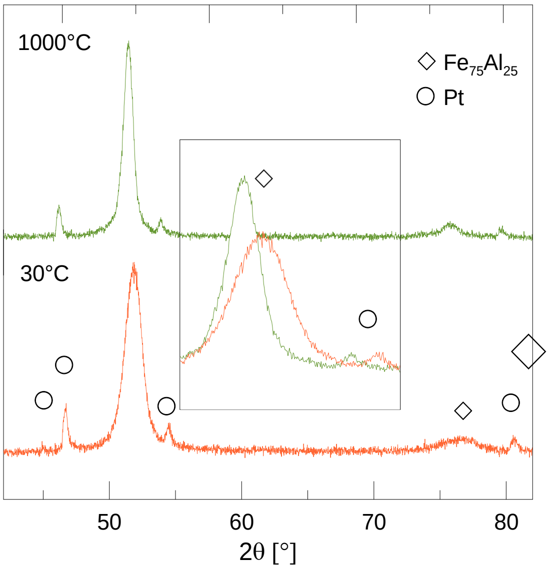

Figure 2. X-ray pattern milled (1920 min) and annealed sample.

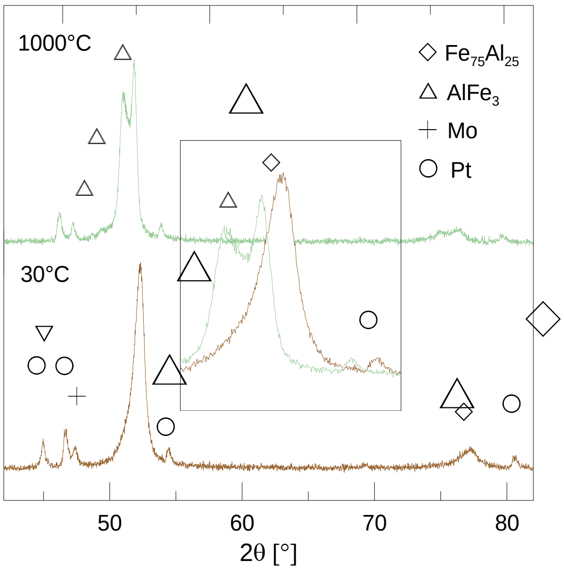

Figure 3. X-ray pattern milled (3840 min) and annealed sample.

Magnetic moment is growing up due to time of milling. The thermomagnetic curves (Fig.4) confirm the decomposition of solid solution to the α-Fe and another magnetic compounds in the samples milled for 960 and 3840 minutes. The magnetic moment of solid solution Fe-Al (sample milled 1920 minutes) was drastically increased after heat treatment due to homogenization of sample and the particle growth.

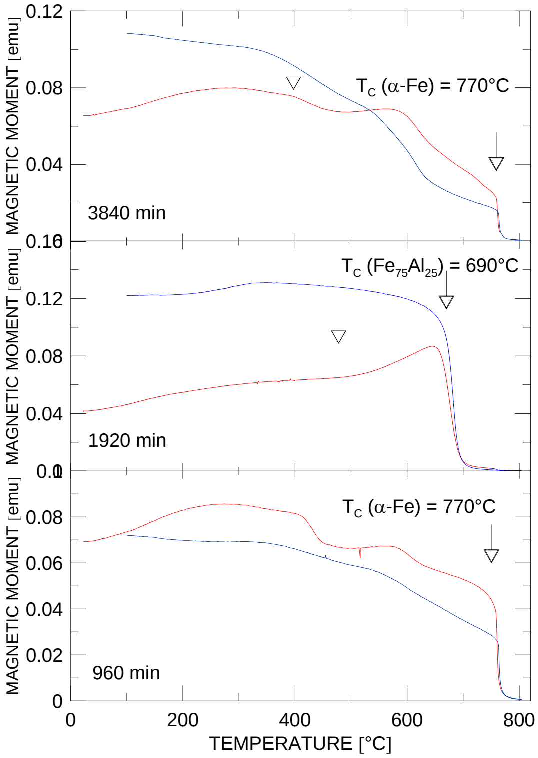

Figure 4. Temperature dependence of magnetic moment.

Conclusion

The thermal stability of alloy did not influenced negatively by the small amount of Mo in the Fe-Al system. The material is comprised of one phase (Fe-Al solid solution) milled 1920 minutes and is stable up to 1000°C. To compare with the pure system (Fe-Al) [1] is the magnetic saturation of this material higher.

References

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