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Temperature dependence of magnetisation of intermetallic compound $GdFe_{11}Ti$ in different structural states

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Abstract

The temperature dependence of magnetisation $\sigma(T)$ and the structure of $GdFe_{11}Ti$ intermetallic compound in coarse-grained, microcrystalline, and nanocrystalline states are investigated experimentally. It is found that thermal stability of $GdFe_{11}Ti$ compound depends on grain size. The phase composition of the coarse-grained sample remains unchanged upon heating to 1100 K. Microcrystalline powder with a grain size of ~1 μm undergoes partial decomposition and transformation into iron phase during grinding and upon heating in the temperature range 800–1100 K. By contrast, heating of bulk nanocrystalline sample up to 1100 K partially restores phase composition. © 2001 Elsevier Science. All rights reserved

Keywords: Magnetization; Nanocrystalline structure; Intermetallic compounds

1. Introduction

The intermetallic compounds $RFe_{11}Ti$ ($ThMn_{12}$-type lattice) were found to be promising, hard magnetic materials, due to high magnetic crystalline anisotropy and adequate Curie point. It is a matter of general experience that magnetic properties of ferromagnets are sensitive to their structure so it is important to study the influence of the structure on both the magnetic properties and the thermal stability, in order to take advantage of this potential. Presently it is known that an increase in lattice parameters as a result of nitrogenation and hydrogenation causes a rise of both magnetisation and Curie point in $RFe_{11}Ti$ compounds [1]. The decrease of the grain size leads to the change of spin reorientation temperature in $DyFe_{11}Ti$ compound [2]. In the present work, we report our recent research on temperature dependence of magnetisation and structure of $GdFe_{11}Ti$ samples. The samples investigated revealed three different microstructures: coarse-grained, microcrystalline, and nanocrystalline.

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2. Results and discussion

Coarse-grained samples, prepared by induction melting under argon atmosphere were initial ones. Microcrystalline powder sample was produced by mechanical attrition of coarse-grained specimen in protecting environment. Nanocrystalline bulk sample was produced, by severe plastic deformation on Bridgman anvils under a pressure of 8 GPa from the initial coarse-grained sample, at room temperature. Scanning electron microscopy (SEM Philips XL30 with EDX system was used) of coarse-grained sample revealed multiphase, non-homogeneous structure with phase area sizes in the range of 10–200 μm, while nanocrystalline samples have homogeneous structure and contain only one phase. Microcrystalline powder consists of particles with mean diameter value about 1 μm. Phase composition analysis of the samples provided by X-ray diffraction techniques (X-ray diffractometer Philips PW17/10 was applied) revealed impurities of α-Fe and Fe_2Ti in the coarse-grained state (Fig. 1a) and only main $GdFe_{11}Ti$ phase in microcrystalline and nanocrystalline (Fig. 1b) states. Significant broadening of lines on X-ray pattern of nanocrystalline sample is caused obviously by a small

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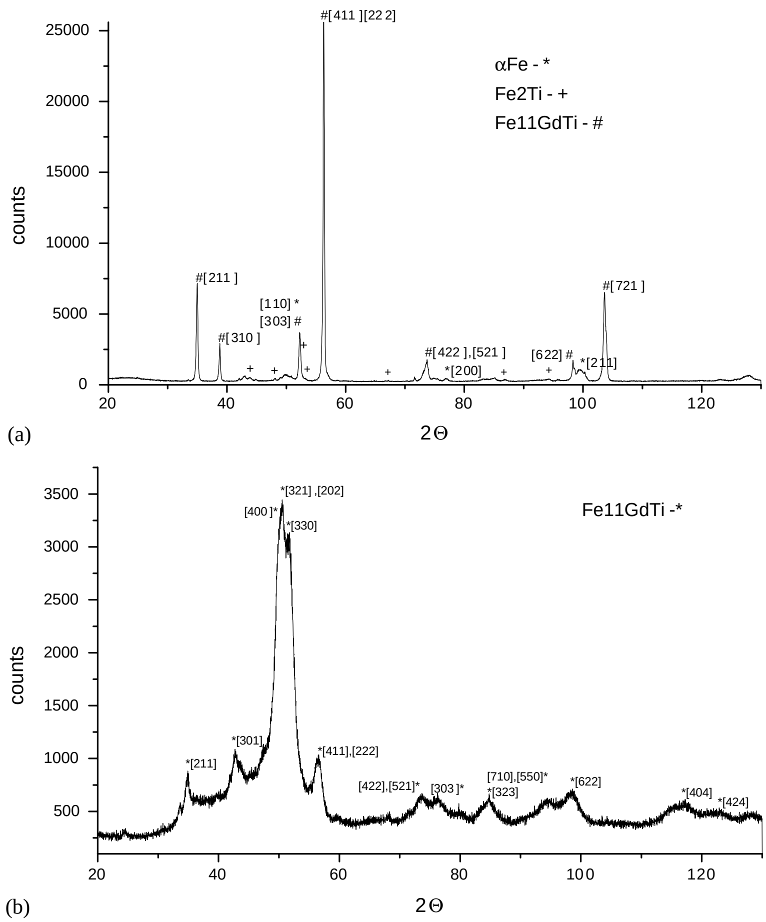

Fig. 1. X-ray diffraction profiles of coarse-grained (a) and nanocrystalline (b) samples of $GdFe_{11}Ti$ compound.

grain size. The small grain size was confirmed by transmission electron microscopy (JEM 2000EX was used) of nanocrystalline sample which revealed crystallite size of about 50 nm.

Temperature dependence of magnetisation $\sigma(T)$ was recorded by automated magnetic balance under the vacuum of 1.3×10^{-2} Pa, for a field of 240 kA/m, in the temperature range 293–1100 K. Two well-defined steps on the curve $\sigma(T)$ for coarse-grained state, corresponding to spin reorientation transition at 500 K and Curie point at 600 K of $GdFe_{11}Ti$ compound, were observed (Fig. 2a). On the similar curves $\sigma(T)$ for nanocrystalline (Fig. 2b) and microcrystalline samples (Fig. 2c) the steps are less pronounced and one more step appears near 1050 K, which is close to the Curie point of pure iron, which indicates partial decomposition of initial $GdFe_{11}$-Ti intermetallic compound. A comparison of $\sigma(T)$ curves corresponding to heating and cooling of coarse-grained sample reveals some retardation of magnetic transitions. It should be noted that the decrease of the applied field increases the retardation. In the case of nanocrystalline sample the curve corresponding to cooling passes lower than the curve recorded during heating, indicating some recovery and ordering of the structure during heating, in the temperature range 1000–1100 K (Fig. 2b). Microcrystalline powder sample exhibits low thermal stability (Fig. 2c). Gradual increase of magnetisation on heating above 800 K and high level of magnetisation on cooling indicates on irreversible decomposition of the initial compound and appearance of pure iron.

The reason for partial decomposition of microcrystalline sample is associated with an interaction of well-developed powder surface with a residual gas. Similarly, detailed investigation of $Er_{0.45}Ho_{0.55}Fe_2$ compound powder during heating in the similar conditions revealed increased reactivity of rare-earth metal ions and formation of oxides and nitrides in different valent states [3]. Under the same condition of measurements such a decomposition was not observed neither in coarse-grained nor in nanocrystalline samples of $GdFe_{11}Ti$. Thus, thermal stability of $GdFe_{11}Ti$ compound depends

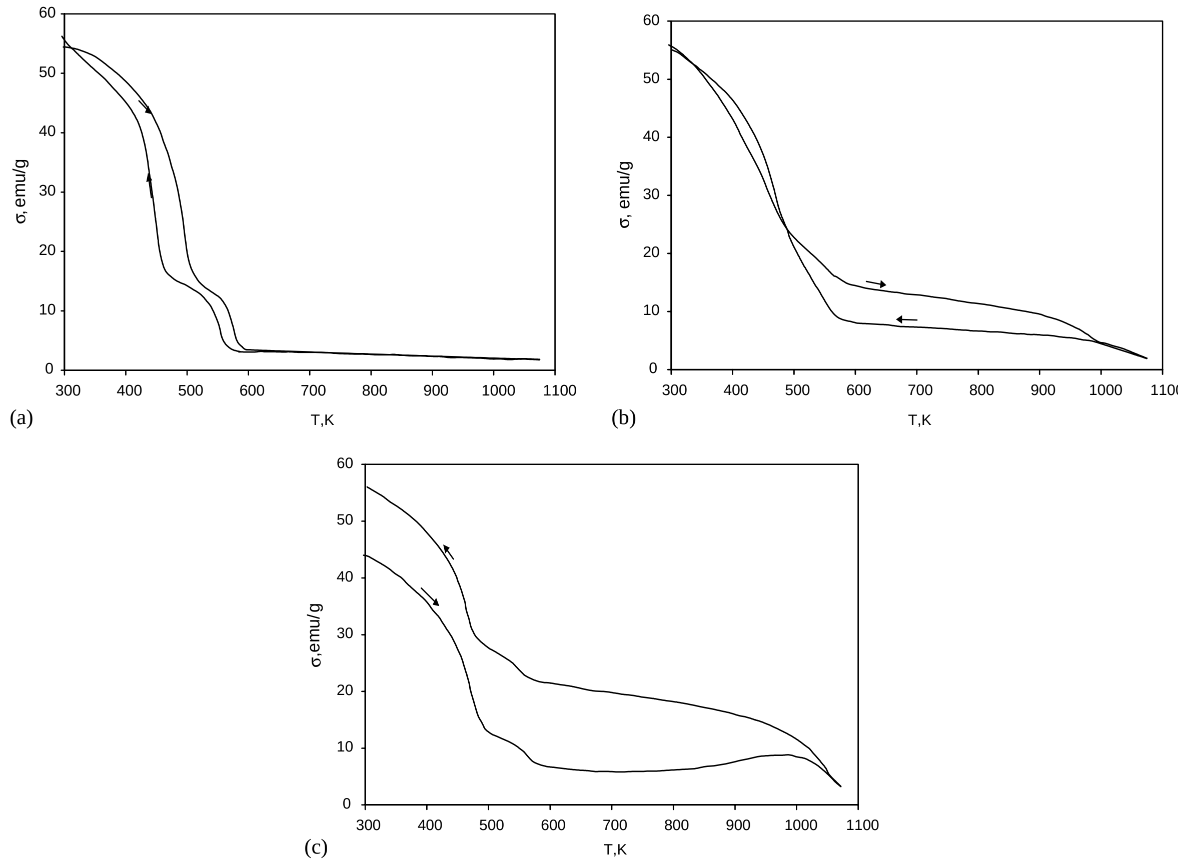

Fig. 2. The temperature dependence of magnetisation of GdFe11Ti compound in coarse-grained (a) nanocrystalline (b) and submicrocrystalline (c) states.

on crystalline grain size and sample preparation technique.

Acknowledgements

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