Journal of Magnetism and Magnetic Materials 401 (2016) 525–531

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

journal homepage: www.elsevier.com/locate/jmmm

Current Perspectives

A study of the physical properties of single crystalline Fe_5B_2P

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ARTICLE INFO

Article history:
Received 21 August 2015
Received in revised form
19 October 2015
Accepted 22 October 2015
Available online 24 October 2015

Keywords:
Single crystal
Magnetization
Demagnetization factor
Arrott plot
Transition temperature
Anisotropy constant

ABSTRACT

Single crystals of Fe_5B_2P were grown by self-flux growth technique. Structural and electrical and magnetic anisotropic properties are studied. The Curie temperature of Fe_5B_2P is determined to be 655 ± 2 K. The saturation magnetization is determined to be 1.72 μ_B/Fe at 2 K. The temperature variation of the anisotropy constant K_1 is determined for the first time, reaching ~0.50 MJ/m^3 at 2 K, and it is comparable to that of hard ferrites. The saturation magnetization is found to be larger than the hard ferrites. The first principle calculations of saturation magnetization and anisotropy constant are found to be consistent with the experimental results.

1. Introduction

The existence of the ternary Fe_5B_2P phase was first reported in 1962 [1,2]. The first reference reported the detailed structural information and the Curie temperature for the Fe_5B_2P phase. The second reference reported the structural details using X-ray and chemical analysis. Its structural prototype is tetragonal Cr_5B_3 with the space group D_{4h}^{l5} – $I4/mcm$. The Curie temperature was reported to fall between 615 K and 639 K, depending upon the B content. In 1967, another study reported a Curie temperature of 628 K and a saturation magnetization of 1.73 μ_B/Fe [3]. The Fe_5B_2P phase was also studied using Mössbauer spectroscopy and X-ray diffraction in 1975 [4]. In addition to confirming the Curie temperature range as well as the average saturation magnetic moment per Fe atom, the Mössbauer study identified the average moment contributed by each of the Fe lattice sites in the Fe_5B_2P unit cell. The Fe(2) (or 4c) sites contribute 2.2 μ_B/Fe. The Fe(1) (or 16l) sites contribute 1.6 μ_B/Fe. The average extrapolated moment of the both sites at 0 K was reported to be 1.73 μ_B/Fe.

Fe_5B_2P is specifically interesting as a possible high transition temperature, rare earth free, hard ferromagnetic material. Given that all prior work on Fe_5B_2P was made on polycrystalline samples, we developed a single crystal growth protocol, measured thermodynamic and transport properties of single crystalline samples, and determined the magnetic anisotropy of this material. The anisotropy constant K_1 is positive, indicating that the c-axis is the easy axis of magnetization, and has a comparable size and temperature dependence as hard ferrites such as $SrFe_{12}O_{19}$ and $BaFe_{12}O_{19}$.

2. Experimental details

2.1. Crystal growth

As part of our effort to search for new, or poorly characterized ferromagnetic compounds, we have developed single crystal growth protocols for transition metal rich, chalcogenide and pnictide binary and ternary phases. In a manner similar to some of our earlier transition metal – sulphur work [5,6], we started by confirming our ability to contain Fe–P binary melts in alumina crucibles sealed in amorphous silica ampules. As outlined by Canfield and Fisk [7] and Canfield [8], sealed ampoules were decanted after slow cooling by use of a centrifuge. Crucibles with alumina filters [9,10] were used to allow assessment and even reuse of the decanted liquid. For this experiment, a mixture of freshly ball milled iron powder and red phosphorous lumps were placed in an alumina crucible in an atomic ratio of Fe:P = 0.83:0.17. A homogenous liquid exists at 1060 °C (i.e. there was no crystal

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http://dx.doi.org/10.1016/j.jmmm.2015.10.088
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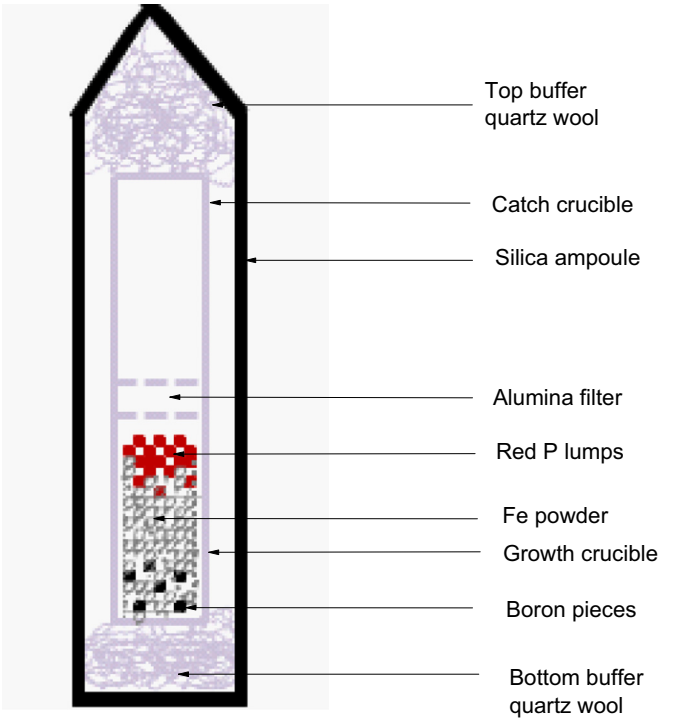

Fig. 1. A schematic assembly of the crystal growth ampoule.

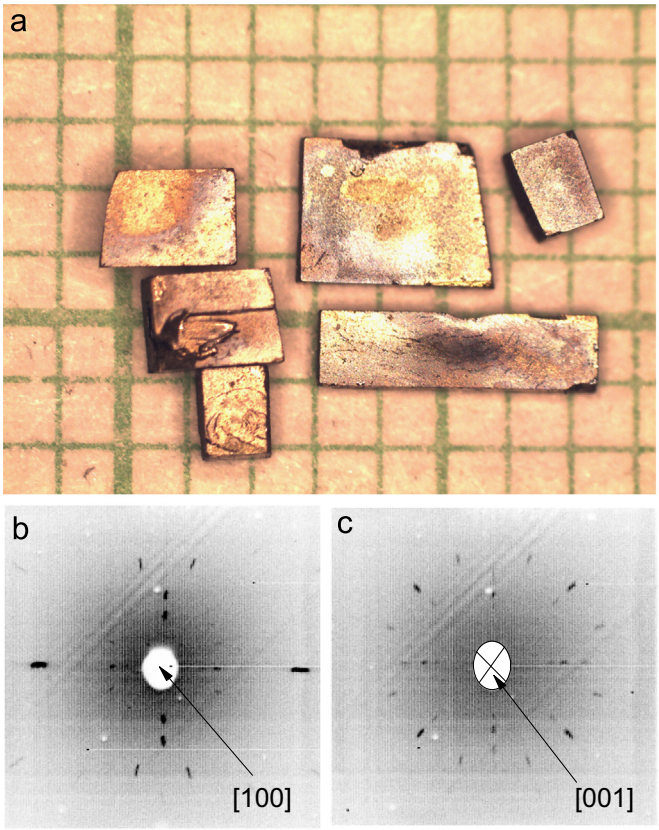

Fig. 2. (a) The acid etched single crystals image of Fe_5B_2P, (b) Laue pattern along the hard axis [100] and (c) Laue pattern along the easy axis [001] of magnetization.

growth upon cooling from 1200 °C to 1060 °C and all of the material decanted). For similar temperature profiles, an initial melt of $Fe_{0.86}P_{0.14}$ leads to the growth of dendritic Fe whereas for initial melts of $Fe_{0.81}P_{0.19}$, $Fe_{0.79}P_{0.21}$ and $Fe_{0.77}P_{0.23}$ faceted Fe_3P was grown. These data are all consistent with the binary phase diagram [11] and indicate that the Fe–P binary melt does not have a significant partial pressure of phosphorous and does not react with alumina.

After some optimization, an initial stoichiometry of $Fe_{72}P_{18}B_{10}$ was used to grow single phase Fe_5B_2P plates. Ball milled Fe (Fe lumps obtained from Ames lab), red phosphorous lumps (Alfa Aesar, 99.999% (metal basis)), and crystalline boron pieces (Alfa Aesar, 99.95%) were placed in an alumina crucible/filter assembly, sealed in a partial pressure of Ar in an amorphous silica tube (as shown schematically in Fig. 1). The ampoule was heated over 3 h to 250 °C, remained at 250 °C for 3 h, heated to 1200 °C over 12 h, held at 1200 °C for 10 h, and then cooled to 1160 °C over 75 h. After cooling to 1160 °C the ampoule was decanted using a centrifuge and plate like single crystals of Fe_5B_2P could be found on the growth side of the alumina filter. In order to confirm that the growth of crystals took place from a complete liquid, we decanted one growth at 1200 °C, instead of cooling to 1160 °C, and indeed found all of the material decanted.

After growth, single crystals were cleaned by etching in a roughly 6 molar HCl solution. Fig. 2(a) shows a picture of the etched single crystals.

2.2. Physical properties measurement

The crystal structure and lattice parameters of Fe_5B_2P were determined with both single crystal and powder x-ray diffraction (XRD). The crystal structure of Fe_5B_2P was determined from single-crystal XRD data collected with the use of graphite monochromatized MoK_α radiation ($\lambda = 0.71073$ Å) at room temperature on a Bruker APEX2 diffractometer. Reflections were gathered by taking four sets of 360 frames with 0.5° scans in ω, with an exposure time of 25 s per frame and the crystal-to-detector distance was 5 cm. The measured intensities were corrected for Lorentz and polarization effects. The intensities were further corrected for absorption using the program SADABS, as implemented in Apex 2 package [12].

For powder XRD, etched single crystals of Fe_5B_2P were selected and finely powdered. The powder was evenly spread over the zero background single crystal silicon wafer sample holder with help of a thin film of Dow Corning high vacuum grease. The powder diffraction pattern was recorded with Rigaku Miniflex diffractrometer using copper K_α radiation source over 8.5 h (at a rate of 3 s dwell time for per 0.01° to cover the 2θ value up to 100°).

To identify the crystallographic orientation of the single crystal plates, Laue diffraction patterns were obtained using a Multiwire Laboratories, Limited spectrometer. The resistivity anisotropy data were measured in a four-probe configuration using a Quantum Design Magnetic Property Measurement System (MPMS) for temperature control and the external device control option to interface with a Linear Research, Inc. ac (20 mA, 16 Hz) resistance bridge (LR 700). To make the resistivity anisotropy data more reliable, resistivity bar for both [100] and [001] axes were taken from the same crystal as shown in the inset of Fig. 5.

The sample preparation for magnetization measurements is a major step in a magnetic anisotropy study. The etched crystal was cut into a rectangular prismatic shape and the dimensions were determined with a digital Vernier caliper.

Temperature and field dependent magnetization was measured using the MPMS up to room temperature and a Quantum Design Versalab Vibration Sample Magnetometer (VSM) with an oven option for higher temperature ($T < 1000$ K). In MPMS, plastic straw was used to align the sample in desired directions. The sample was glued to the VSM sample heater stick with Zircar cement obtained from ZIRCAR Ceramics Inc. While gluing, the sample was pushed into the thin layer of Zircar paste spread on the heater stick to