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Magnetisation of bulk $Mn_{11}Si_{19}$ and Mn_4Si_7

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ABSTRACT

The purpose of this paper is to determine by experiment whether $Mn_{11}Si_{19}$ and Mn_4Si_7 in their bulk states have a finite magnetic moment or not. Magnetisation measurements were carried out on these materials using both SQUID system and Kerr rotation system. The high quality samples were grown using the temperature gradient solution growth method. SQUID measurements revealed that $Mn_{11}Si_{19}$ has finite magnetism while Mn_4Si_7 does not in their bulk states. It was also confirmed that Mn_4Si_7 became magnetic and $Mn_{11}Si_{19}$ got to exhibit a distinctive hysteresis in their powdery state. The enhancement of magnetism implied that the surface of the samples was to a great extent linked to its magnetism.

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1. Introduction

Higher manganese silicides (HMSs) ($MnSi_x$, with x ~1.7) have attracted attention because of their potential as the key material in a future opto-spintronics technology [1]. The new technology exploits three properties of the material, i.e., conventional charge electronic, optical, and magnetic properties. The HMSs are highly anticipated to serve as a material for the three properties to play on. There are several different phases of HMSs with similar crystal structures such as Mn_4Si_7, $Mn_{11}Si_{19}$, $Mn_{14}Si_{26}$, and $Mn_{27}Si_{47}$. Despite similarity in atomic structures, it has not yet been clarified whether the band structure and magnetic properties are also similar. It is important to understand such characteristics associated with the different phases, for HMSs to materialise as an opto-spintronics material.

In this paper, we focus on the magnetism of $Mn_{11}Si_{19}$ and Mn_4Si_7. SQUID system and optical Kerr effect measurement system were used for the magnetisation measurements. First we determine whether $Mn_{11}Si_{19}$ and Mn_4Si_7 have finite magnetism in their bulk states or not. This issue has recently been debated both theoretically and experimentally [3–5], but there has been no decisive argument on it. For example, Gottlieb et al. concluded by experiment that Mn_4Si_7 has magnetism [3]. Migdas et al. concluded by numerical calculations that $Mn_{11}Si_{19}$ has magnetism and Mn_4Si_7 has no magnetism [4]. Yabuuchi et al. concluded by numerical calculations that in bulk crystal there is no magnetism for either $Mn_{11}S_{19}$ or Mn_4Si_7 [5]. It is therefore important to determine experimentally the magnetisation of those crystals.

The second purpose of this paper is to compare the magnetism between the bulk and powdery states in $Mn_{11}Si_{19}$ and Mn_4Si_7. Yabuuchi et al. pointed out that finite magnetisation emerges in nano-particles of those material embedded in host silicon, because of strain effect, charging effect, and interface effect [5]. Motivated by this study, we explore the magnetism in powdery state.

To carry out those purposes, we first have to prepare crystals of $Mn_{11}Si_{19}$ and Mn_4Si_7 with single-phase. This is because we anticipate that the discrepancy in the magnetism of those materials in previous works [3–5] originate from the presence of several phases of HMSs in single samples. In fact, it has been difficult to synthesise single-phase crystals of them. To overcome this difficulty, we use the temperature gradient solution growth [6]. We synthesise Mn_4Si_7 and $Mn_{11}Si_{19}$ using this method and it is confirmed by X-ray diffraction that these crystals are single crystals. It has been further confirmed that Mn_4Si_7 is a semiconductor while $Mn_{11}Si_{19}$ is a metal [7].

2. Experiment

To prepare bulk crystals of Mn_4Si_7 and $Mn_{11}Si_{19}$, the temperature gradient solution growth method [6] was used. Fig. 1 displays a schematic diagram of highly purified quarts ample (right) and a temperature profile (left). Ga or Sn solvent is used. The growth temperature is 870 °C, and the source temperature is between 880 and 940 °C. The temperature gradient is 50 °C/cm. The growth time is 168 h.

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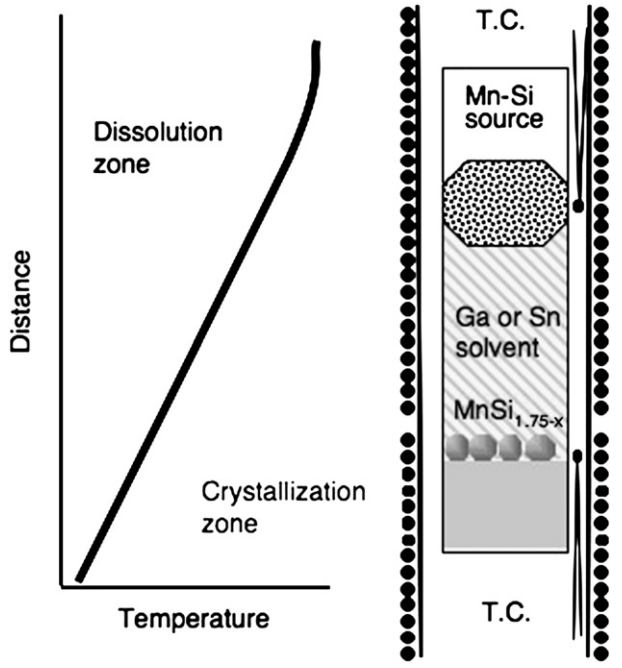

Fig. 1. The temperature gradient solution growth method.

Magnetisation measurements were carried out on these two samples both in their bulk and in their powdery states, using two methods. The two methods are a commercial SQUID system (Magnetic Property Measurement System (MPMS) MultiVu, QuantumDesign) and our Kerr rotation measurement system.

In the SQUID system, the sample was gently fixed in a plastic capsule by filling inside the capsule with Teflon tapes until the sample was fixed. The capsule was, then, fitted in a plastic straw. Finally, the straw was attached to the edge of a metal rod, which enters the cryostat of the MPMS system. The data were all taken for various magnetic fields between −50,000 and 50,000 Oe, under temperatures from 5 to 290 K. The weight of the samples $Mn_{11}Si_{19}$ and Mn_4Si_7 in their bulk and powdery states are 42 and 34 mg (bulk) and 24 and 6 mg (powder), respectively. The magnetisation of a sample measured in emu is divided by its weight, to get the normalised magnetisation. The values of magnetisation for a capsule without a sample in it, at a corresponding temperature, were subtracted from the raw data, to get the background free one. The subtraction precedes the normalisation process mentioned above.

In our Kerr rotation measurement system, the samples were located on a Cu sample stage, which is cooled down to 4 K. The principle of the system involves, through variation in its optical properties, detecting variation in magnetic states of the sample under magnetic fields. Variation in the angle of polarised light between incident and reflected lights corresponds to that in the sample's magnetic moment. Measurements were performed with magnetic fields being perpendicular to the stage for between −3000 and 2000 Oe. Variation in the magnetisation is expressed in that of grey values associated with the reflected light. With a rotation analyser for a polarised light being fixed at some angle, the variation in the angle of the reflected light is to be reflected to that in its intensity, which is scaled in grey values from 0 to 4096. Our Kerr system can be considered to have a spatial resolution of about 1 μm owing to optics employed in the system.

Specific procedures, as to powdery samples, for obtaining a grey value for a certain grain under a magnetic field are as follows: Firstly, pound a bulk crystal in a mortar to obtain a powdery sample. We obtained powdery samples of them with each grain size of about 20–40 μm for $Mn_{11}Si_{19}$ and 60–80 μm for Mn_4Si_7 (Fig. 2). During the pounding, Mn_4Si_7 is softer and easier to break than $Mn_{11}Si_{19}$. Secondly,

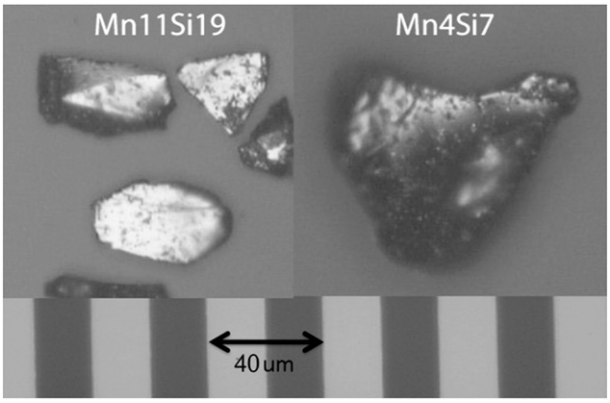

Fig. 2. $Mn_{11}Si_{19}$ and Mn_4Si_7 in their powdery states. By pounding using a mortar, powdery samples with each grain size of 20–40 μm and 60–80 μm are obtained for $Mn_{11}Si_{19}$ and Mn_4Si_7, respectively.

locate a certain amount of powder for either of the two samples, on a single Cu stage using double-stick tape and cool the stage down to 4 K. Thirdly, apply a certain amount of magnetic field over whole the samples on the stage. Fourthly, shine a single grain by an incident light, which is polarised, and measure the intensity of a reflection light from the surface of the grain. Since a polarisation angle of the reflection light varies in accordance with magnetic moment induced by a magnetic field, the intensity of the reflection light through an eyepiece of a microscope varies. For the rotation analyser is set before the eyepiece so that, for example, the reflection light should have a lowest intensity when there is no variation between the polarisation angles of incident and reflection lights. In this case, for example, variation of magnetic moment in the sample corresponds to brightness of the reflection light. Fifthly, integrate a grey value over a certain area and regard the value as representing the whole area. In our experiment, dimensions of the integration area are about 10 μm × 10 μm.

3. Results and discussions

Figs. 3 and 4 show magnetisation measurements, by the SQUID system, in their bulk states. As mass of the Mn_4Si_7 sample is smaller than that of the $Mn_{11}Si_{19}$ sample, the noise in the magnetisation data is larger in the Mn_4Si_7 sample than in the $Mn_{11}Si_{19}$ sample. Fig. 3

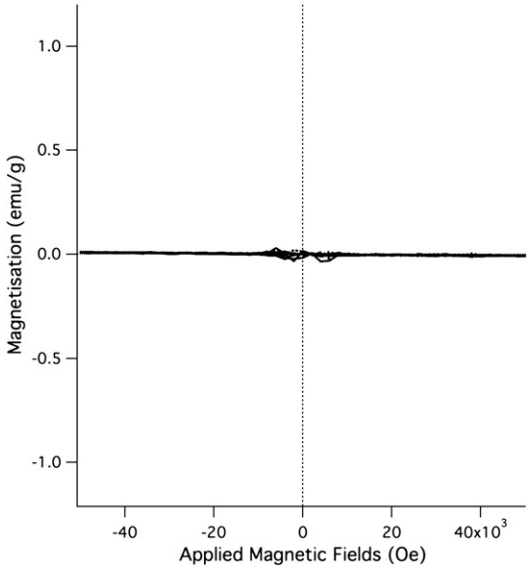

Fig. 3. Magnetic field dependence of the magnetisation of Mn_4Si_7 in the bulk state (by SQUID).

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