Anomalous Hall effect in β -FeSi₂

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(Received 1 July 1998)

The dependence of the Hall resistivity ρ_H on the magnetic induction *B* was investigated on β -FeSi₂ single crystals and molecular-beam-epitaxy-grown layers. The strength of anomalous contributions to $\rho_H(B)$ found in most samples critically depends on the growth conditions. Measurements of the magnetization *M* were performed on single crystals. Measurements of $\rho_H(B)$ and M(B) were performed on the same sample. It is shown that the anomalous contributions to $\rho_H(B)$ have a magnetic origin. The measurements of *M* and the results of neutron diffraction gave no indication of a magnetic phase transition of β -FeSi₂. It is suggested that the cause of the anomalous contributions to the Hall resistivity found in most samples investigated is of extrinsic nature. We propose that the effects arise from clusterlike regions which carry large magnetic moments and behave superparamagnetically. [S0163-1829(98)06047-0]

I. INTRODUCTION

 β -FeSi₂ belongs to the group of semiconducting silicides. This semiconductor found special interest due to the direct gap at about 0.87 eV,¹ which suggests applications combining optoelectronics and Si technology. An example of a Si LED using β -FeSi₂ precipitates in the active region showing electroluminescence at a wavelength of 1.5 μ m was demonstrated recently.² Other applications have been proposed in the field of thermoelectrics.³

For all fields of applications a thorough knowledge of the electrical properties of the material is necessary. Investigation of the resistivity and the Hall coefficient can yield valuable information on transport properties such as the concentration and the mobility of the free carriers. However, interpretation of Hall data of β -FeSi₂ has been complicated by nonlinear dependences of the Hall resistivity ρ_H on the magnetic induction B especially at low temperatures.^{4–6} Hysteresis effects of the Hall resistivity have been reported, too.^{7,8} Interpretation of these anomalous contributions to the Hall effect are discussed controversially in the literature. On the one hand, the effects are believed to be caused by a contribution of an anomalous Hall effect due to the transition of β -FeSi₂ to a magnetic phase at low temperatures.^{4,8} On the other hand, nonlinear dependences of ρ_H on B for n-type samples have been explained by a two-band model in which the free electrons have different effective masses.⁶ This interpretation excludes the contribution of an anomalous Hall effect.

General results of our resistivity and Hall effect measurements on β -FeSi₂ samples are reported elsewhere.⁹ In this paper we report on the investigations of the dependence of the Hall resistivity on the magnetic field in the temperature range 12–300 K on a variety of β -FeSi₂ single crystals prepared by chemical vapor transport (CVT) and of thin layers deposited with molecular-beam epitaxy (MBE). In addition, magnetization measurements were performed on individual single crystals. Neutron diffraction experiments were carried out to look for a magnetic phase in β -FeSi₂. We will show that (i) there is a magnetic cause of the nonlinear behavior of $\rho_H(B)$ and (ii) it is probable that nonstoichiometric regions carrying large magnetic moments and behaving like superparamagnetic cluster are the cause of the nonlinear dependence of $\rho_H(B)$.

II. EXPERIMENT

The single crystalline samples were grown by chemical vapor transport (CVT) using iodine as a transport agent. The starting material was introduced into a silica ampoule. The evacuated ampoule was inserted horizontally into a two-zone furnace with a temperature gradient T_1 - T_2 . T_1 is the temperature in the dissolution zone and T_2 is the temperature in the crystallization zone. The growth time was typically 200 h. Single crystals were grown at Hahn-Meitner-Institut (Lab 1), the University of Konstanz (Lab 2), and the Institut für Festkörper- und Werkstofforschung, Dresden (Lab 3). A survey of the samples investigated is given in Table I. For further details, see Ref. 10, 11, and 12. The CVT-made crystals showed needlelike shapes with lengths of up to 10 mm and diameters of up to 1 mm. The needles were investigated by x-ray diffraction. For all further investigations only needles were used that were in the semiconducting β phase.

Thin layers were grown by molecular-beam epitaxy (MBE) using a template technique on *n*-type Si(111) substrates having a resistivity of 5 k Ω cm. All layers were grown under the same conditions by codeposition of Fe and Si in stoichiometric quantities at a temperature of 660 °C. The purity of the Si source was 5*N*. The purity of the Fe source material varied between 99.95% (Goodfellow) and 99.9985% (Alfa). The thickness of the layers ranged between 200 and 350 nm. All layers showed *p*-type conduction in the temperature range *T*<200 K where the influence of the silicon substrate on the electrical measurement is negligible.⁹

For electrical measurements, six-point arrangements of contacts were attached to the crystals directly by a Pt adhesive or by soldering with In. Layers were cut into $5 \times 5 \text{ mm}^2$ pieces and Al dots in the corners provided a van der Pauw configuration. The contacts were proved to be Ohmic by coplanar IV measurements in the whole temperature range. The dependence of the Hall resistivity on the magnetic induction was measured with a sweep technique in

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TABLE I. Growth conditions and characterization of the samples used for the experiments. An asterisk denotes purity of Si source $\geq 5N$ for all samples, except for needles grown from FeSi powder (indicated by a dagger symbol).

Sample	Grown in Lab.	Doping element	<i>n</i> - or <i>p</i> -type	$T_1 - T_2 ^{\circ} C$ (CVT only)	purity Fe source*	$ \rho_H(B) $ nonlinear	hysteresis of $\rho_H(B)$
N1	2		n	1000-800	$\geq 3N$	yes	yes
N2	2	Cr	р	1050-800	$\geq 3N$	yes	no
N3	1	Р	n	1000 - 800	5N	yes	no
<i>N</i> 4	1	Mn	р	1000 - 800	3 <i>N</i>	yes	yes
N5	1		n	1000 - 800	5N	yes	yes
N6,N7,N8	1	Cr	р	950-680	$2N^{\dagger}$	yes	yes
N9	1		р	950-680	$2N^{\dagger}$	yes	yes
N10	3		n	1000 - 750	5N	yes	yes
N11	3	Cr	р	1050 - 750	5N	yes	no
N12	3	Co	n	1050 - 750	5N	yes	no
L1	(layer)		р		99,95%	yes	no
<i>L</i> 2	(layer)		р		99,9985%	no	no

the temperature range 12–300 K with fields of up to 0.8 T. The measured voltage V_M consists of several signal contributions:⁸

$$V_{M} = V_{OS} + V_{I} + V_{MR} + V_{H}.$$
 (1)

From V_M the contributions due to the misalignment of the Ohmic contacts (V_{OS}) and the contribution induced inductively by the time-varying magnetic field (V_I) were subtracted. The remaining signal V_B consists of the Hall voltage V_H and the voltage due to the change of the magnetoresistance V_{MR} . In the β -FeSi₂ samples, a small negative magnetoresistance arises only at very low temperatures ($T < 50 \text{ K}, \Delta V_M / V_M < 0.5\%$ at 0.6 T) and could mostly be neglected. If V_{MR} was significant, the separation of the two signals was achieved using the following formulas:

$$V_{\rm MR} = 0.5 [V_B(B_0) + V_B(-B_0)], \qquad (2)$$

$$V_H = 0.5 [V_B(B_0) - V_B(-B_0)].$$
(3)

The magnetization was measured with a commercial SQUID magnetometer (Quantum Design). The measurements were performed on single needles in order to enable comparison with electrical measurements on the same sample. We used a special (diamagnetic) sample holder and fixed the sample with commercial adhesive (UHU Hart), which is known to cause only little magnetic background. The samples were measured in magnetic fields between ± 1 T in the temperature range 15–300 K. Only one branch of the hysteresis loop was measured (from +1 T to -1 T). Due to the long delay time after each change of the value of the magnetic field and the long averaging time due to the small value of the sample signal, each measurement for one fixed temperature value took about 12 h. The background signal from the sample holder and the adhesive was measured in a separate run for each temperature and subtracted from the data. The diamagnetic parts of the sample signal were subtracted from the data, too. For the analysis of the ferromagnetic parts of the data, the following equation which describes a hysteresis curve was used:

$$y(B) = B_s/2 - \frac{M_s}{1 + \exp\left(\frac{B + B_c}{dB}\right)}.$$
 (4)

In this expression M_S denotes the value of saturation, B_C the value of the coercive force, and dB the width of the curve.

Neutron diffraction experiments were performed at the diffractometer E6 of the BER II reactor of the Hahn-Meitner-Institut, Berlin. Powder diffraction patterns were taken in an angular range from 5° to 84°. The incident neutron wavelength was 2.4 Å. The powder was ground from about 40 needles (Lab 1) grown under similar conditions without intentional doping. The samples came from charges where investigated needles showed hysteresis of $\rho_H(B)$ (N9 in Table I).

III. RESULTS

The dependence of the Hall resistivity $\rho_H = V_H / I$ on the magnetic induction $B[\rho_H(B)]$ for the samples N1, N2, and N3 at 15 K is shown in Fig. 1. N1 is representative for samples which show a pronounced hysteresis of the Hall resistivity at low temperatures (coercive force $B_C \approx 24$ mT at 15 K). The hysteresis is strongly dependent on temperature and vanishes around 150 K. N2 and N3 exhibit nonlinear dependence without hysteresis. At 60 K, two samples (N2 and N3) show linear dependence of the Hall resistivity on the magnetic field, while sample N1 shows a hysteresis effect with a coercive force $B_C \approx 2 \text{ mT}$ (Fig. 2). The general observation is that nonlinear dependences and hysteresis effects of $\rho_H(B)$ vary from sample to sample. The sign of the anomalous contribution is positive regardless of whether the sign of the normal Hall coefficient is positive (N2) or negative (N3) at higher temperatures. Samples with a hysteresis of $\rho_H(B)$ at low temperatures still show a nonlinear dependence of the Hall resistivity in the temperature range where a hysteresis of Hall resistivity is no longer noticeable. In some of these samples $\rho_H(B)$ was nonlinear up to room temperature. Apparently the hysteresis of the Hall resistivity can



FIG. 1. Hall resistivity ρ_H as a function of the magnetic induction *B* for the single crystalline samples *N*1, *N*2, and *N*3 at 15 K. Note the different range of the magnetic induction for sample *N*3 (inset).

merely be considered to be the result of a particularly pronounced nonlinear dependence.

The nonlinear dependence of $\rho_H(B)$ was also observed in some of the MBE-grown layers at low temperatures (Fig. 3). However, the effect is orders of magnitude smaller in these samples (*L*1) than in single crystals. In the case of sample



FIG. 2. ρ_H as a function of *B* for the samples *N*1, *N*2, and *N*3 at 60 K.



FIG. 3. ρ_H as a function of *B* for two MBE-grown layers (*L*1,*L*2). Layer *L*1 was grown with starting material of lower purity than in the case of layer *L*2.

L2, $\rho_H(B)$ shows the regular linear dependence down to the lowest temperatures. No anomalous contribution to the Hall resistivity was found in this layer. The samples L1 and L2 were grown under the same conditions but using different Fe sources. The purity of the Fe source was 99.98% for sample L1 and 99.9985% for sample L2 (purity of Si source 5N for both samples). This suggests that the purity of the starting material may influence the strength of the anomalous contributions to $\rho_H(B)$. However, some slight changes of the growth conditions due to, e.g., a possible instability of the Fe sources cannot be excluded.

The results of the measurements of $\rho_H(B)$ for various samples are summarized in Table I. They suggest that the existence and the strength of a nonlinear behavior of $\rho_H(B)$ are not exclusively correlated with the purity of the starting material but that other details of the growth process such as the temperature regime of the CVT process also play an important role.

In order to address the question of whether anomalous contributions to $\rho_H(B)$ are related with magnetic effects, magnetization measurements were performed on single crystals taken from a growth ampoule with Cr-doped crystals which showed hysteresis effects in the Hall resistivity (see Table I). Figure 4(a) shows results of magnetization measurements for a single needle (*N*7) at a temperature of 60 K. Note that the dominant diamagnetic part of the signal was subtracted as described above. The magnetization shows a small hysteresis with $B_C \approx 7$ mT. A fit to the data using formula (4) is shown, too. The parameters of the fit curve were $M_S = 0.0115$ emu/cm³, $B_C = 6.7$ mT, and dB = 36 mT. In order to obtain an estimate of the density of the magnetic moments we used the equation

$$M_{S} = g \mu_{B} S N, \tag{5}$$



FIG. 4. Magnetization *M* as a function of the magnetic induction *B* for the single crystalline sample *N*7 at 60 K (a). Temperature dependence of the coercive force B_C for the hysteresis loops of Hall resistivity (\bullet) and magnetization (\Box) measured on sample *N*8 (b).

with N being the concentration of the contributing centers, g the Landé factor, S the spin, and μ_B Bohr's magneton. With the value M_s from the fit, 1 emu $\approx 1.1 \times 10^{20} \mu_B$ and g = 2, we obtained a value of 4×10^{17} cm⁻³ for NS. Hence N should be in the range 10^{17} – 10^{18} cm⁻³. This value is far below the atomic density and also considerably smaller than the concentration of the Cr acceptor which was determined to be $\geq 1 \times 10^{19}$ cm⁻³ by our Hall-effect measurements. The result shown in Fig. 4(a) suggests that the anomalous contributions to $\rho_H(B)$ are related to the magnetization of the samples. We were able to prove this suggestion directly by measuring both magnetization and Hall effect in the temperature range 15-100 K on the same sample. The specimen used (N8) exhibited pronounced hysteresis phenomena in $\rho_H(B)$ and in M(B) at low temperatures. In Fig. 4(b) we use the coercive force B_C to represent a characteristic feature of these hysteresis curves since B_C is the only parameter of a magnetic hysteresis loop which is not influenced by the demagnetization factor. Figure 4(b) demonstrates that B_C for the $\rho_H(B)$ and the M(B) hysteresis loops exhibits the same temperature dependence in the range 15-100 K. It is important to notice that no extra adjustment of the two sets of data has been applied. The comparison of the two curves in Fig. 4(b) leads us to the conclusion that the observed hysteresis of the Hall resistivity is directly related to the magnetization of the samples.

It has been suggested in the literature that anomalous contributions to $\rho_H(B)$ might be related to a magnetic phase transition of β -FeSi₂.^{4,8} Although the above results contradicted the assignment of these effects to an intrinsic property of the samples, we performed neutron scattering in order to look for a possible magnetic phase transition of the material.



FIG. 5. Neutron diffraction measurements for undoped β -FeSi₂ at 200 and 1.6 K. An offset was added to the 200 K data set.

Two data sets measured at 1.6 and 200 K are shown in Fig. 5. No additional reflection could be observed in the 1.6 K spectrum in comparison with the spectra taken at 200 K. From the difference in the peak intensities we could not determine a contribution of a magnetic phase in the 1.6 K data set. From these observations we have to conclude that no intensity contributions due to a magnetic phase are present in the 1.6 K spectrum within the signal-to-noise ratio.

Previous investigations suggested that for FeSi₂ samples showing hysteresis effects of $\rho_H(B)$, the remnant Hall voltage $V_H(B=0)(t)$ was not stable.⁸ We performed measurements of the time dependence of the Hall voltage at zero field for sample N8 with strong hysteresis effects at low temperatures. After applying a magnetic field of 0.5 T, we measured the Hall voltage as a function of time well outside the magnetic field. Figure 6 shows the decay of the remnant Hall voltage $V_H(B=0)(t)$ at 12 K. The baseline of the measurement was determined from an extrapolation to long times $[V_H(B=0)(t\to\infty)]$. It is important to note that only a portion of the remnant Hall voltage relaxes such that a residual signal remains, which appears to be stable at the time scale of these measurements. We estimated that about 50% of the initial $V_H(B=0)$ decays at this temperature. The inset of Fig. 6 shows that the decay is exponential with a time constant $\tau \approx 9.8$ s. An increase of the temperature to 21 K led to a strong decrease of τ .

IV. DISCUSSION

The different behavior of the various samples indicates that anomalous contributions to $\rho_H(B)$, i.e., hysteresis effects and nonlinear dependences, are not caused by an intrinsic property of the β -FeSi₂. On the contrary, there are several factors which influence the occurrence and strength of



FIG. 6. Time decay of the remnant Hall voltage of sample N8 for two different temperatures. The baseline was determined by $V_H(B=0)(t\to\infty)$. The decay is exponential (inset). Note that only about 50% of the initial $V_H(B=0)(t=0)$ decays.

these effects. Comparing the investigated samples, it becomes obvious that the purity of the starting material is an important factor. MBE layers grown with an Fe source of purity 99.95% do show nonlinear dependence of the Hall resistivity at low temperatures, while all layers grown with the purer Fe source (99.9985%) under otherwise unchanged conditions show no anomalous contributions to the Hall resistivity down to 15 K. The effect of the purity of the starting material can also be seen to some extent in the case of single crystalline needles grown by the CVT technique. Generally the samples grown with a starting material of high purity showed only small anomalous contributions to the Hall resistivity which vanished at temperatures not far above 15 K. Samples with strong anomalous contributions, i.e., hysteresis of the Hall resistivity and nonlinear dependency up to high temperatures, are usually the samples grown with starting material of poor purity.

However, the purity of the starting material is not the only factor which influences the strength of the anomalous contributions. Our results suggest that in the case of the CVT crystals the temperature regime during growth plays a role, too. For example, only some samples from lab 2 (e.g., N1) and from lab 3 (e.g., N10) showed hysteresis effects. They were grown at a temperature T_1 of 1000 °C in the dissolution zone. On samples grown at $T_1 = 1050$ °C (e.g., N2, N11, and N12), hysteresis effects were not observed. In the case of lab 1 in which generally lower temperatures were used, most of the samples showed hysteresis effects. They were most pronounced in the samples N6-N8 grown at the lowest temperatures (950-680 °C). Thus it seems that CVT crystals made with 5N material and at a temperature in the dissolu-

tion zone of at least 1050 °C show only small anomalous contributions to the Hall resistivity and no hysteresis effects.

We thus conclude that the observed anomalous contributions to the Hall resistivity in the β -FeSi₂ samples are caused by deviations from the ideal crystal structure rather than by intrinsic properties of the material. These deviations are influenced by several factors, including the purity of the starting material and the temperature regime of the CVT process. An influence of additional factors cannot be excluded. An optimized growth process should lead to β -FeSi₂ samples which show no anomalous contributions to the Hall resistivity.

Up to now, hysteresis effects in β -FeSi₂ crystals were only reported by Teichert *et al.*^{7,8} In these works, crystals from Lab 1 were investigated. Hysteresis behavior was reported for all the samples at low temperatures. At room temperature, nonlinear dependences of $\rho_H(B)$ were only found in *n*-type samples.⁸ Our results obtained from a broader spectrum of samples show that the type of conduction plays no role. On the one hand, we have found *p*-type single crystals showing a nonlinear dependence of $\rho_H(B)$ up to room temperature (*N*4). On the other hand, there are single crystals in which we found only small anomalous contributions to $\rho_H(B)$ and no hysteresis down to 15 K (*n*-type, *N*3; *p*-type, *N*6). We conclude that the type of conduction and the strength of the anomalous contributions to $\rho_H(B)$ are unrelated.

Models which assume that band-structure effects are the cause of the observed nonlinear dependences in *n*-type crystals⁶ are not consistent with the experimental results. Our experiments prove that anomalous contributions to $\rho_H(B)$ are related to the magnetization of the sample and therefore have to be interpreted with the additional contribution of an anomalous Hall effect.

For ferromagnetic materials the following empirical relation is generally used to describe the additional contribution of an anomalous Hall effect to $\rho_H(B)$ (in SI units).¹³

$$\rho_{(H)}(B) = R_H B + R_A M. \tag{6}$$

In this formula, R_H denotes the normal Hall coefficient, R_A the anomalous Hall coefficient, and M the magnetization. Valassiades et al.4 and Teichert8 explain anomalous contributions to $\rho_H(B)$ in their samples as being due to the additional contribution of such an anomalous Hall effect. They propose that this anomalous Hall effect is related to a magnetic phase transition of the β -FeSi₂ to a ferromagnetic and antiferromagnetic phase, respectively. Our results, on the other hand, prove that β -FeSi₂ shows no phase transition to a magnetic phase in the relevant temperature range. The ferromagnetic signal in our magnetization measurements on individual needles is of the order 10^{-3} emu/cm³, which is in agreement with the order of magnitude that was reported for magnetization measurements on several single crystals.⁶ This signal is only a small contribution to the total magnetic momentum of the sample. The main contribution being diamagnetic is proportional to the field. The additional ferromagnetic contributions are thus by far too small to be caused by a ferromagnetic phase of the β -FeSi₂ sample.⁶ Moreover, we have found no hint of a magnetic phase transition in the temperature-dependent measurements of $\rho_H(B)$ and M(B)

or in the neutron scattering experiment. EPR measurements performed by Irmscher *et al.* on β -FeSi₂ needles also gave no indication of a magnetic phase transition.¹⁴ Szymanski *et al.* performed measurements of the magnetic susceptibility on FeSi_{2-x}Al_x down to 4.2 K with magnetic fields up to 5 T.¹⁵ The samples were produced with iron of relatively low purity (99.95%). The authors reported that they found no indication of a magnetic phase transition but observed in some cases weak ferromagnetic signals which saturated at fields lower than 1 T and had a transition temperature between 100 and 150 K. The authors interpreted these additional signals by some inhomogeneities and speculated that they are caused by precipitates of iron-rich Fe-Si particles behaving like superparamagnetic clusters.¹⁵

We suggest that the observed small ferromagnetic signals in the magnetization and the anomalous contribution to the Hall resistivity are caused by inhomogeneities in the samples. The assumption of superparamagnetic clusters can explain important details of our experimental observations. The relaxation effect shown in Fig. 6 is a strong indication of the existence of superparamagnetism in our samples. Superparamagnetism is characterized by a relaxation of the remnant magnetization which accelerates with increasing temperature.¹⁶ The time constant τ describing this relaxation is also extremely sensitive to the size of the superparamagnetic particles. By definition, particles are considered to be stable, i.e., ferromagnetic, if $\tau > 100$ s. The temperature at which $\tau = 100$ s is called the blocking temperature. Observations of the anomalous Hall effect due to the presence of superparamagnetic clusters are not unusual in materials which contain atoms carrying large magnetic moments. For example, in a recent investigation of In_{0.82}Mn_{0.18}As/Al_{0.3}Ga_{0.7}Sb heterostructures, nonlinear $\rho_H(B)$ behavior and, at low temperatures, hysteresis and relaxation of the remnant magnetization were found.17 The authors ascribed these observations to clusters of Mn atoms behaving superparamagnetically.

Being related to inhomogeneities, it is reasonable to assume that the superparamagnetic clusters in our samples have a size distribution. In this interpretation the relaxation of Fig. 6 arises from clusters with a blocking temperature well below 12 K. Clusters of larger size are responsible for the residual contribution of the remnant magnetization. With increasing temperature the number of superparamagnetic clusters which are still stable shrinks. This explains the gradual decrease of the strength of nonlinear effects in $\rho_H(B)$ [see Fig. 4(a)].

V. SUMMARY

In summary, anomalous contributions to the Hall resistivity in β -FeSi₂ are not caused by an intrinsic property of the material but by deviations from the ideal crystal structure. The strength of the anomalous contributions sensitively depends on the growth conditions. The optimization of such parameters as the purity of the starting material and the temperature regime of the CVT process reduces or completely quenches these anomalous contributions to $\rho_H(B)$. In the magnetization measurements we found small ferromagnetic signals. By measuring the hysteresis of $\rho_H(B)$ and M(B) on the same sample, we proved that anomalous contributions to $\rho_H(B)$ have a magnetic origin. Neutron scattering gave no hint of a magnetic phase transition of the β -FeSi₂. The observed anomalous contributions to $\rho_H(B)$ can be explained by the assumption of superparamagnetic clusters in our samples.

ACKNOWLEDGMENTS

The authors would like to thank Ch. Kloc and G. Behr for the preparation of some of the single crystals as well as P. Stauss for the preparation of the MBE layers.

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