Magnetotransport and magnetocaloric effects in intermetallic compounds
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Chapter 3
Experimental

3.1 Sample preparation

Polycrystalline samples were prepared by arc-melting the pure starting materials in a water-cooled copper crucible in a continuously Ti-gettered Ar atmosphere. The purity of the starting materials is at least 99.9%. Usually, the surface of the starting materials was cleaned by etching in an appropriate way described in ref. 3.1. The batches were turned over and remelted several times to achieve good homogeneity. In order to further improve the homogeneity and in order to reduce stresses, the buttons were wrapped in Ta foil and annealed in special water-free quartz ampoules. For each compound, the annealing temperature and duration of the heat treatment is given in the relevant chapters.

Part of the results described in this thesis was obtained by measurements on single-crystalline samples of (Hf,Ta)Fe$_2$ and Gd$_5$(Ge$_2$Si$_1$)$_4$. The single-crystalline samples were grown with the travelling-floating-zone method in an adapted NEC double-ellipsoidal-type image furnace. The furnace consists of two ellipsoidal mirrors which are gold plated. The filaments of two halogen lamps are positioned in the focus of each of the mirrors, and are projected on the common focal point of the two mirrors. In this way, a certain input power is concentrated on the melt zone between the feed and the seed. The temperature of the melt zone is controlled by the dc-voltage of the two lamps. The design of the image furnace and the details of the growth process are described extensively by Hien [3.2] and Duijn [3.3].

The feeds were prepared by arc-melting the pure starting materials into a button, which was then cast into a cylindrical rod of 4 mm diameter. The feeds, with compositions Hf$_{0.80}$Ta$_{0.20}$Fe$_{2.05}$, Hf$_{0.86}$Ta$_{0.14}$Fe$_{2.05}$ and Gd$_5$Ge$_2$Si$_{1.633}$, had an excess of Fe/Si to compensate for evaporation during the growth. A quartz tube with a diameter of 7 cm served as growth-chamber. Before the growth, the quartz tube was pumped vacuum for one night to a pressure of $10^{-6}$ mbar, and then filled with 800 to 900 mbar 99.999 vol.% pure Ar. During the growth, the Ar atmosphere was continuously purified with a TiZr getter. The feed and seed were counter-rotated with typical speeds of 25 rpm. The pulling speed of the shafts was 8 mm/h for the (Hf,Ta)Fe$_2$ crystals and 3 mm/h for the Gd$_5$(Ge$_2$Si)$_4$ crystal. After the growth, the samples were slowly cooled down to room temperature during one night. In this way, three single-crystals were obtained with a diameter of 4 mm and a length of about 7 cm.
3.2 Characterisation techniques

The polycrystalline samples were characterised at room temperature by means of X-ray powder diffraction by using a commercially available Philips PW 1700 diffractometer with Cu-Kα radiation. Of each sample about 0.5 gram was powdered by ball-milling for 3 to 5 minutes. The recorded spectra were analysed by means of the Rietveld refinement procedure [3.4] by using the program FullProf [3.5]. In this way, the lattice parameters are determined with an accuracy of 0.005 Å. Furthermore, the presence of secondary phases of about 5 vol.% can be detected. Some details on the refinement procedure are given in section 3.6 on neutron-diffraction techniques. In addition, of the compounds Fe₃(Ga₀.₉₀Al₀.₁₀)₄ and Gd₅Ge₂₄Si₁₁.₆, X-ray powder diffractograms were measured as function of temperature between 4.2 and 300 K. The temperature was controlled to an accuracy of about 1 % in a home-built ⁴He-gas-flow cryostat.

The quality of the single-crystalline samples was checked by means of the X-ray backscattering Laue technique. The radiation of a W anode was used as an incident beam and the reflections were recorded on a Polaroid photographic film. The crystals were oriented by using the program OrientExpress [3.6], which generates from a few given reflections the whole Laue pattern. An example of the recorded and simulated Laue patterns of the Hf₀.₈₆Ta₀.₁₄Fe₂ single crystal is shown in figure 3.1.

The homogeneity and the stoichiometry of most of the compounds were checked by means of electron-probe micro-analysis (EPMA) by Mr. Gortenmulder at the Kamerlingh

**Figure 3.1.** Recorded and simulated X-ray Laue patterns of a Hf₀.₈₆Ta₀.₁₄Fe₂ single crystal oriented along the b' axis.
Onnes Laboratory, University of Leiden [3.7]. In the EPMA experiment the sample is exposed to an electron beam with an acceleration voltage of 15 to 20 kV. The electron beam partly ionises the different elements of the compound, thus creating vacant energy levels. Electrons with higher energy levels occupy these vacant levels, emitting photons. The photon energy is characteristic for the energy-level difference and hence for the elements. The number of photons with this characteristic energy counted per second yields a measure for the concentration of atoms of a certain type when it is compared to standard intensities. With EPMA, variations in the homogeneity of the sample can be detected with an accuracy better than 1%. The absolute accuracy is limited to 3%, depending on the element and the standard used. Furthermore, with the EPMA equipment scanning electron microscopic images of the surface can be made, yielding information on the grain size and the distribution of possible secondary phases.

3.3 Electrical-resistivity measurements

The electrical resistance $R$ was measured by means of the standard four-point method with a low excitation Linear Research LR-700 ac-resistance bridge. The bridge operates at a frequency of 16 Hz and can measure resistances from $2 \, \text{M}\Omega$ to $2 \, \text{m}\Omega$ with an accuracy of $2 \times 10^{-5}$. Lower resistances can be measured down to n\Omega. The LR-700 uses a four-point ac lock-balance technique to measure the resistance of a sample. Two leads are used to apply a fixed-amplitude ac current to the sample. Two additional leads send the resultant voltage back to the bridge to be balanced against an equal and opposite ac voltage. The value of the opposite ac voltage gives the value of the sample resistance. The ac four-point technique eliminates errors that might be caused by lead resistance or sample contact resistance. Furthermore, it eliminates dc-voltage errors.

The electrical resistivity $\rho$ is obtained from the electrical resistance $R$ by

$$\rho = R \frac{A}{l}$$

where $A$ is the cross section of the sample perpendicular to the current direction, and $l$ is the distance between the voltage contacts. The samples were shaped by means of spark-cutting into rectangular bars with typical dimensions of $1 \times 1 \times 4 \, \text{mm}^3$. The precise determination of $A$ and $l$ was done by using a calibrated microscope. The absolute accuracy in $\rho$ is only 10 to 20% due to errors in the determination of $A$ and $l$, and due to the possible presence of microscopic cracks and holes in the sample. In case the absolute value of $\rho$ could not be determined, the resistivity curves have been normalised to the room-temperature value.

The current and voltage contacts were realised by four different methods. Most of the contacts were prepared by soldering Cu wire of 0.1 mm thickness with In-based alloys as a solder and a saturated ZnCl$_2$ solution as a flux. For a good result, it is important to set the
temperature of the soldering iron only about 10 to 20 degrees higher than the melting point of the solder. In a later stage of the research, a dc-resistance welding system of Unitek Equipment was purchased. With this system, wires can be welded on the samples while controlling the applied current and voltage. For our samples, the input power used was of the order of 100 W, while the pulse length was typically 6 to 10 ms. In case the above two methods were not successful, the contacts were prepared with silver paint. Besides, the electrical resistivity of single-crystalline Gd₅Ge₂.₄Si₁.₆ samples was measured by pressing the contacts onto the samples.

The temperature dependence of the electrical resistivity between 4.2 and 300 K was measured in home-built equipment. The temperature of the sample was determined by either a carbon-glass or a cernox thermometer with an accuracy better than 1%. The typical cooling/heating rate of the temperature scans was 50 mK/s. The electrical resistivity in magnetic fields was measured with a home-built insert placed in a Quantum Design MPMS cryostat. The applied magnetic field can be varied from −5 to 5 T, the temperature range extending from 1.7 to 400 K. The samples were oriented either parallel or perpendicular to the magnetic field with an accuracy of a few degrees. The experimental set-up for dc-magnetoresistance measurements in the Amsterdam high-field installation is described in the next section.

3.4 Magnetic measurements

The macroscopic magnetic properties of the compounds were investigated in a variety
of experimental set-ups. In this section, the measuring technique of the SQUID (Superconducting QUantum Interference Device) magnetometer is described and the specifications of the high-field installation of the University of Amsterdam are given.

At the Van der Waals-Zeeman Institute, an MPMS2-type SQUID magnetometer of Quantum Design has been installed. The temperature range for the measurements extends from 1.7 to 400 K and the applied magnetic field produced by a superconducting magnet, ranges from −5 to 5 T. The SQUID utilises an extremely sensitive detection method that is capable of measuring magnetisation values in the range of $10^{-12}$ to $10^3$ Am$^2$ with an accuracy of 0.1%. The detection system comprises sensing loops, a superconducting transformer with a radio-frequency-interference (RFI) shield, and the SQUID sensor itself with its control electronics. A scheme of the SQUID sensing loops and input circuit is shown in figure 3.2. The basic element of a SQUID sensor is a ring of superconducting metal containing one or more weak links coupled to a tuned circuit driven by a radio-frequency (RF) current source. When a magnetic flux is applied to the ring, an induced current flows round the superconducting ring. In turn, this current induces a periodic variation of the RF voltage across the circuit. A feedback arrangement is used to minimise the current flowing in the ring, the size of the feedback current being a measure of the applied magnetic flux. For a detailed treatise on the operation principles of RF SQUID sensors we refer to ref. 3.8.

The detection loops are configured as a highly balanced second-derivative coil set with a total length of approximately 3 cm. The coils are designed to reject the uniform field of the superconducting magnet to a precision of about 0.1 %. The normal measurement process is to position the sample below the detection coils and then to raise the sample stepwise through the coils. At each step, the output voltage of the coils is measured with the SQUID

![Figure 3.3. SQUID output response to a sample scan.](image)
High-field magnetisation and magnetoresistance measurements were performed in the high-field installation at the University of Amsterdam [3.9], which is capable of generating pulsed fields up to 40 T. Within the constraints of the equipment, the shape of the field pulse can be controlled in any desired way. Two types of magnetic-field pulses were used for the measurements presented in this thesis (figure 3.4). For the magnetisation measurements, a stepwise pulse was used to measure at quasi-static fields. At each step, the field is kept constant for at least 50 ms to minimise the influence of eddy currents in the sample. After a settling time of about 20 ms, the maximum deviation of the actual field from the desired constant field is less than about 5 mT. The magnetisation of the sample is measured by an inductive method with a relative accuracy of about 0.5 %. The pick-up coil system can detect magnetic moments as small as $10^{-5}$ Am$^2$.

The second type of pulses was generated by ramping the magnet to the desired field and then short-circuiting it with a series inductance. Here the field-decay rate is not controlled, but is completely determined by the self-inductance and the resistance of the circuit. This type of pulse was used for magnetoresistance measurements as the mechanical vibrations of the lead wires is avoided as much as possible in this way. The field dependence of the electrical resistance was measured with a four-point dc-method. The samples were oriented either parallel or perpendicular to the magnetic field with an accuracy of a few degrees. Both the magnetisation and the magnetoresistance measurements were performed at 4.2 K on samples immersed in liquid $^4$He.

![Figure 3.4. Typical field pulses generated in the high-field installation of the University of Amsterdam. The step-wise pulse is used for magnetisation measurements; the free-decay pulse is used for magnetoresistance measurements.](image-url)
3.5 Measurements under hydrostatic pressure

Ac-susceptibility and thermal-expansion measurements under hydrostatic pressures up to 10 kbar were carried out on Fe$_3$(Ga,Al)$_4$ samples, in a collaboration with Prof. dr V. Sechovský and dr K. Prokeš at the Department of Metal Physics, Charles University, Prague [3.10]. The pressure cell is made of a CuBe alloy, with oil serving as pressure-transmitting liquid. The cell is mounted on a cooling head in a Leybold closed-cycle refrigerator system. The pressure is determined in situ by electrical-resistivity measurements of a calibrated Mn sensor. A Micro-Measurements SK-350 strain gauge is used for the thermal-expansion measurements. For the ac-susceptibility measurements, two coils of 50 windings each were wound around the rectangular-shaped samples with dimensions $2 \times 3 \times 4$ mm$^3$. Upon application of an ac magnetic field

$$H = H_0 e^{i\omega t}$$

(3.2)

with angular frequency $\omega$ via the primary coil, the magnetisation $M$ of the sample will be

$$M = M_0 e^{i(\omega t - \delta)}$$

(3.3)

where $\delta$ is a phase delay. The complex ac susceptibility $\chi_{ac}$ then reads

$$\chi_{ac} = \frac{M}{H} = \frac{M_0}{H_0} \cos \delta - i \frac{M_0}{H_0} \sin \delta = \chi' - i \chi''$$

(3.4)

The real (in phase) component $\chi'$ is connected with the reversible initial magnetisation process. The value of the imaginary (out of phase) component $\chi''$ represents the energy loss during the initial magnetisation process [3.11]. The ac susceptibility $\chi_{ac}$ was determined by measuring the inductance voltage of the secondary coil using a lock-in amplifier operating at a frequency of 90 Hz. The measurements of the real component of the ac susceptibility as a function of temperature were used to determine the magnetic transition temperatures of the Fe$_3$(Ga,Al)$_4$ compounds.

3.6 Neutron-diffraction experiments

With the advent of nuclear reactors, thermal-neutron scattering has become a powerful technique to study the microscopic properties of condensed matter. The usefulness of thermal neutrons arises from the following four properties of the neutron [3.12; 3.13]:

1. The wavelength of thermal neutrons is comparable to the interatomic distances in solids (1
to 3 Å), so that interference effects may occur which yield information on the structure of the solid.

2. The neutron is uncharged and hence can penetrate deeply into the solid. Furthermore, as there is no Coulomb barrier to overcome it can approach the nuclei closely.

3. The neutron has a magnetic moment ($\mu_n = 1.913 \mu_N$), which implies that neutrons interact with the unpaired electrons in magnetic atoms so that it is possible to probe the magnetic structure of the solid.

4. The energy of thermal neutrons (about 25 meV) is of the same order as many excitation energies in matter. Therefore, inelastic neutron scattering provides information on the elementary excitations and the dynamics of the solid on an atomic scale.

Thus, the scattering of a neutron in a solid is dominated by two types of interaction: the interaction between the neutron and the nuclei, and the interaction between the magnetic moment of the neutron and the magnetic moments in the solid. In this section, we will briefly describe the procedure to obtain information on the structural and magnetic properties of a solid from elastic neutron diffraction. For an extensive treatise we refer to ref. 3.14.

The scattering process of a neutron by an atom in a solid is described by an incoming state characterised by a wave vector $k_0$ and a spin $\sigma_0$, and an outgoing state characterised by a wave vector $k_1$ and a spin $\sigma_1$. The differential scattering cross section $d\sigma/d\Omega$ for elastic scattering is given by

$$\left( \frac{d\sigma}{d\Omega} \right)_{k_0,\sigma_0\rightarrow k_1,\sigma_1} = \left( \frac{m_n}{2\pi\hbar^2} \right)^2 |\langle k_1,\sigma_1 | V_{\text{int}} | k_0,\sigma_0 \rangle|^2$$

where $\Omega$ is a solid angle and $V_{\text{int}}$ is the interaction potential between the neutron with mass $m_n$ and the atom in the solid. Note that $\sigma$ and $\sigma_0$, $\sigma_1$ represent different quantities. For elastic scattering $|k_0| = |k_1|$. The scattering vector of the process is given by $Q = k_0 - k_1$. For elastic scattering of an unpolarised beam of neutrons, the total scattering cross section is the superposition of a nuclear and a magnetic contribution. Interference effects between these two contributions vanish as they contain the average of the neutron-spin direction [3.15]. In the following, we will consider the nuclear and the magnetic contribution to $d\sigma/d\Omega$.

As the size of the nuclei (about $10^{-5}$ Å) is much smaller than the neutron wavelength (1 to 3 Å), the nuclear contribution to the interaction potential can be described within the Born approximation by the so-called Fermi pseudo-potential. For a set of nuclei forming a solid, the pseudo-potential $V(r)$ is

$$V(r) = \frac{2\pi\hbar^2}{m_n} \sum_j b_j \delta(r - r_j)$$

where the sum extends over a unit cell containing $j$ nuclei with scattering length $b_j$, at rest at positions $r_j$. The scattering length $b_j$ has a value of the order of $10^{-15}$ m, the exact value
Figure 3.5. Variation of the neutron-scattering length as a function of atomic weight [3.16].

Varying irregularly as a function of element number (figure 3.5). Combination of equations 3.5 and 3.6 yields for the nuclear differential cross section \( \frac{d\sigma}{d\Omega} \)

\[
\left( \frac{d\sigma}{d\Omega} \right) = \left| \sum_j b_j \exp(iQ \cdot r_j) \exp(-W_j) \right|^2 = \frac{(2\pi)^3}{V_{cell}} \sum_{\tau} |F_N(Q)|^2 \delta(Q - \tau) \quad (3.7)
\]

where \( \exp(-W_j) \) is the square root of the Debye-Waller factor, which accounts for the thermal motion of the atoms around their equilibrium position, \( V_{cell} \) is the volume of the unit cell and \( \tau \) are those positions at the reciprocal lattice where the Bragg law holds. \( F_N(Q) \) is termed the nuclear structure factor. In fact, \( |F_N(Q)|^2 \) is proportional to the measured intensity of a nuclear reflection. The set of reflections, together with their positions determined by the Bragg law, contain all structural information (crystal structure, lattice parameters, position and type of nuclei, Debye-Waller factors, etc.) of the investigated solid.

The magnetic-scattering cross section \( \frac{d\sigma}{d\Omega} \)_M is more complex to analyse since the relevant magnetic interactions are of vector nature. A similar relation holds for both the magnetic and the nuclear interaction potential (equation 3.6). Here, the constant nuclear scattering length \( b_j \) should be replaced by a \( Q \)-dependent magnetic-scattering length \( a_j(Q) \)

\[
a_j(Q) = -\gamma_0 \sigma \left[ \hat{Q} \times (s_j \times \hat{Q}) + \frac{i}{\hbar |Q|} (p_j \times \hat{Q}) \right] \quad (3.8)
\]
where $\gamma_0 = 2.695 \times 10^{-15}$ m, $\sigma$ is the spin of the scattered neutron, $s_j$ and $p_j$ are the spin and momentum of electron $j$, respectively, and $\hat{Q} = Q/|Q|$ is the unit scattering vector. The right-hand-side of equation 3.8 consists of two terms: an interaction term due to the spin part and an interaction term due to the orbital part of the magnetic moment of the unpaired electrons. Evaluation of equations 3.5, 3.6 and 3.8 yields for the magnetic differential cross section

$$
\left( \frac{d\sigma}{d\Omega} \right)_M = \left[ \sum_j \gamma_0 m_{\perp j} f_j(Q) \exp(iQ \cdot r_j) \exp(-W_j) \right]^2 = \frac{(2\pi)^3}{V_{\text{cell}}} \sum_q \sum_\tau |F_M(Q)|^2 \delta(Q-q-\tau)
$$

where $m_{\perp j} = \hat{Q} \times (m_j \times \hat{Q})$ is the projection of the magnetic moment $m_j$ of atom $j$ onto the scattering plane defined by $\hat{Q}$ and $f_j(Q)$ is the magnetic form factor of atom $j$. $F_M(Q)$ is termed the magnetic structure factor and $q$ is the magnetic-propagation vector.

Let us now consider the results obtained so far. From equations 3.7 and 3.9 it becomes clear that both nuclear and magnetic interactions contribute to the neutron scattering. Note that $\gamma_0$ is of the same order of magnitude as $b_j$. Hence, both nuclear and magnetic scattering have a comparable contribution to the total scattering. For a perfect crystal, the intensity due to nuclear scattering corresponds to delta functions at positions $\tau$ on the reciprocal lattice determined by the Bragg law. In contrast, for a perfect magnetic structure the intensity due to magnetic scattering concentrates on delta functions located at $Q = \tau + q$. Furthermore, nuclear scattering is essentially independent of $Q$ and occurs at all temperatures. Magnetic scattering, on the other hand, decreases sharply with increasing $|Q|$ and is only present below the magnetic-ordering temperature. These differences supply, in principle, sufficient information to discriminate between nuclear and magnetic scattering, and thus to determine the crystallographic and magnetic structure of the solid under investigation.

Usually, the first step in investigating the microscopic magnetic properties of a solid is to perform a neutron-diffraction experiment on a powdered sample. The d-spacings between crystallographic or between magnetic planes in a crystal are determined by measuring the intensity as a function of angle of incidence $\theta$. As mentioned before, the peak positions are given by the well-known Bragg law.

In the framework of the present study, powder-neutron-diffraction experiments were carried out on the (Hf,Ta)Fe$_2$ compounds in the diffractometer E6 at the Hahn-Meitner Institute (HMI), Berlin [3.17]. This diffractometer is equipped with a BF$_3$ banana-type detector with 200 channels of 0.1° width. The detection range is $5^\circ < 2\theta < 115^\circ$. For a neutron wavelength of $\lambda = 2.4$ Å, the neutron flux at the sample position is $5.1 \times 10^6$ neutrons/(cm$^2$/s). Samples with a mass of about 10 gram were mounted in an cryofurnace that is capable of controlling the temperature between 1.5 and 600 K with an accuracy better than 1%. Neutron-powder-diffraction experiments on Fe$_3$Ga$_4$ were performed in collaboration with dr Rodriguez-Carvajal at the Laboratoire Leon Brillouin (LLB), Saclay [3.18].
For further investigation of the magnetic structure of (Hf,Ta)Fe$_2$ compounds, neutron-diffraction experiments were performed on single-crystalline samples. The equipment used is the two-axis diffractometer E4 at the HMI. This diffractometer is equipped with a single-channel detector that covers the range of scattering angle $-110^\circ < 2\theta < 110^\circ$. The temperature range extends from 1.5 to 400 K.

The data obtained by neutron and X-ray diffraction were analysed by means of the Rietveld refinement procedure [3.4], by using the program FullProf [3.5]. The only requirement of the Rietveld refinement procedure is that the structure of the sample is at least approximately known. The whole diffraction spectrum is fitted by using a suitable peak-shape function and by taking as variables the instrumental characteristics (resolution of the detector) and the structural parameters (lattice parameters, position of nuclei, Debye-Waller factors, etc.). The refinement routine minimises the function

$$M = \sum_j w_j (y_j - y_{cj})^2$$  \hspace{1cm} (3.10)

where $w_j$ is the weight assigned to observation $y_j$ (usually $w_j = 1/\sigma_j$ where $\sigma_j$ is the variance), and $y_j$ and $y_{cj}$ are the measured and calculated intensities at the $j$th point of the spectrum, respectively. The calculated intensities $y_{cj}$ are determined by summing the contributions of the Bragg reflections and the background

$$y_{cj} = y_{bj} + \sum_k^{\text{Phases}} S_k \sum_l n_{kl} L_{kl} O_{kl} |F_{kl}|^2 \Omega_{jk}$$  \hspace{1cm} (3.11)

Here, $y_{bj}$ is the background intensity at the $j$th point, $S_k$ is a scale factor of the $k$th phase, $n_{kl}$ is the multiplicity of the $lth$ reflection, $L_{kl}$ is the Lorentz factor, $O_{kl}$ is a correction factor describing preferential orientation, $F_{kl}$ is the structure factor, and $\Omega_{jk}$ is the peak-profile function. The summation $k$ is over all phases and the summation $l$ is over all reflections of the $k$th phase. The quality of the fit is judged by the value of

$$\chi^2 = \frac{M}{N - P + C}$$  \hspace{1cm} (3.12)

where $N$ is the number of data points, $P$ is the number of refined parameters, and $C$ is the number of constraints. The value of $\chi^2$ is expected to converge to a value close to 1 for a successful refinement.
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