Neutron depolarisation experiments on medium carbon steels

F.A.M. Maas

Supervisors:
Ir. S.G.E. te Velthuis
Dr.ir. M.Th. Rekveldt
Dr.ir. J. Sietsma
Prof.dr.ir. S. van der Zwaag

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Delft University of Technology
Laboratory of Materials Science
Section Heat Treatment and Technology
Rotterdamseweg 137
2628 AL Delft

Interfaculty Reactor Institute
Radiation Physics Group
Mekelweg 15
2629 JB Delft
Abstract

A microstructure of a steel is determined by the fraction transformed and the grain size. In principle by doing neutron depolarisation experiments it is possible to monitor both the fraction transformed and the particle size in situ.

The difference in magnetic behaviour between ferrite (ferromagnetic below 773 °C) and austenite and cementite (paramagnetic) is used in neutron depolarisation experiments to measure the ferrite fraction during the entire transformation. The magnetised sample is passed through by a polarised neutron beam. The polarisation vector will precess around the local magnetic induction in every magnetic domain. Also a shortening of the polarisation vector will occur because of the local fluctuations of the magnetic induction (the individual ferrite grains).

In calculating the fraction transformed and the grain size, the magnetisation of the sample is an input parameter. Therefore the magnetisation properties of each component of the microstructure and of the combinations of components that occur during the transformation must be known. By doing experiments with a varying applied external field, information about the magnetisation at each stage of the transformation is obtained. This information is valuable for the interpretation of the experiments.

In the past, several experiments were carried out on C60 (0.60%C) and 57Cr3 [1]. In this work experiments on steels with lower carbon content are presented. These are isochronal experiments on C22 (0.22%C), C35 (0.35%C) and C45 (0.45%C). Lowering the carbon content raises some problems. The temperature at which the ferrite will start to form will be higher. For C22 and C35 the start temperature of the transformation is above the Curie temperature of the ferrite phase, so the initial stage of the transformation could not be measured by this magnetic technique. Isochronal experiments have been carried out at a constant field for all samples. One experiment on C35 has been carried out with a varying applied field. To determine the possible neutron wavelength dependence of the results, sample C35 has also been measured at a smaller wavelength.

In the experiments on C60 and 57Cr3 a peak in the particle size was found at a ferrite fraction of 0.07. In the results presented in this work the same peak has been found in C22, C35 and C45. The position of this peak indicates that it is caused by the microstructure in the early stages of the pearlite transformation.
Symbols

\( \alpha_{ij} \)  elements of the correlation matrix \([\text{mT}^2]\)
\( \alpha_s \)  growth coefficient
\( \delta \)  average particle radius \([\text{m}]\)
\( \delta_{ij} \)  Kronecker delta
\( \gamma \)  gyromagnetic ratio \([\text{s}^{-1}\text{T}^{-1}]\)
\( \gamma_y \)  mean local orientation of \(B(r)\), average of the normalised magnetic induction variation projection on the \(y\)-axis
\( \phi \)  flux \([\text{Wbm}^2]\)
\( \varphi \)  rotation angle \([\text{rad}]\)
\( \varphi_r \)  remanent rotation \([\text{rad}]\)
\( \lambda \)  wavelength \([\text{m}]\)
\( \mu \)  permeability \([\text{Hm}^{-1}]\)
\( \xi \)  correlation parameter \([\text{mT}^2]\)
\( B \)  magnetic induction \([\text{T}]\)
\( \bar{B} \)  local magnetic induction \([\text{T}]\)
\( \langle \bar{B} \rangle \)  mean magnetic induction \([\text{T}]\)
\( B_s \)  spontaneous magnetic induction \([\text{T}]\)
\( c_{eq}^\alpha \)  equilibrium carbon concentration in ferrite
\( c_{eq}^\gamma \)  equilibrium carbon concentration in austenite
\( c_m \)  carbon concentration in austenite
\( c_{111} \)  elastic constant \([\text{Jm}^{-3}]\)
\( D \)  diffusion coefficient \([\text{m}^2\text{s}^{-1}]\)
\( D \)  mean domain width \([\text{m}]\)
\( \tilde{D} \)  depolarisation matrix
\( E \)  energy of a \(180^\circ\) Bloch wall \([\text{Jm}^{-2}]\)
\( f \)  fraction ferromagnetic material
\( H \)  magnetic field \([\text{Am}^{-1}]\)
\( H_c \)  coercivity \([\text{Am}^{-1}]\)
\( H_d \)  demagnetisation field \([\text{Am}^{-1}]\) \(H_d=N_d*M\)
\( I \)  intensity \([\text{counts}]\)
\( K \)  anisotropy constant \([\text{Jm}^{-3}]\)
\( l \)  magnetostrictive constant
\( L \)  mean grain length \([\text{m}]\)
\( L_s \)  sample thickness \([\text{m}]\)
\( L_{sw} \)  transmission length \([\text{m}]\)
\( m \)  magnetisation factor
\( M \)  magnetisation \([\text{Am}^{-1}]\)
\( M_s \)  saturation magnetisation \([\text{Am}^{-1}]\)
\( n \)  number of elementary atomic magnetic dipoles per unit volume
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>$N_d$</td>
<td>demagnetisation factor</td>
</tr>
<tr>
<td>$&lt; n_y^2 &gt;$</td>
<td>average of the square of the $y$-component of the magnetisation</td>
</tr>
<tr>
<td>$p$</td>
<td>magnetic moment</td>
</tr>
<tr>
<td>$\vec{P}$</td>
<td>polarisation vector</td>
</tr>
<tr>
<td>$Q_0$</td>
<td>polarising power of the polariser/analyser combination</td>
</tr>
<tr>
<td>$\hat{R}$</td>
<td>rotation matrix</td>
</tr>
<tr>
<td>$T_c$</td>
<td>Curie temperature [$^\circ$C]</td>
</tr>
<tr>
<td>$T_e$</td>
<td>the temperature at which there is a first deviation in the rotation compared to the rotation above the Curie temperature [$^\circ$C]</td>
</tr>
<tr>
<td>$t$</td>
<td>time [s]</td>
</tr>
<tr>
<td>$t_n$</td>
<td>start time for growth [s]</td>
</tr>
<tr>
<td>$\hat{U}$</td>
<td>unit matrix</td>
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<tr>
<td>$v$</td>
<td>velocity [ms$^{-1}$]</td>
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1 Introduction

The properties of steel are determined by the microstructure and the chemical composition. In the last step of the steel process, the rolling mill and the runout cooling table (the cooling trajectory between the last mill and the coil onto which the steel strip is wound), the microstructure takes shape. Here the transformation of austenite (fcc iron) to ferrite (bcc iron) and pearlite takes place. Depending on the austenite grain size and the cooling rate a certain microstructure is formed. In research on this process, neutron depolarisation experiments enable simultaneous measurement of the degree of transformation and the average particle size. Therefore, these experiments can give insight in the formation processes that determine the final microstructure.

In the neutron depolarisation experiments a polarised neutron beam passes through the sample, which is placed in a furnace. If the sample is in the austenitic phase (paramagnetic) there will be no magnetisation, and nothing will happen to the polarisation vector of the beam. As soon as the ferrite (ferromagnetic below 770 °C) transformation starts, the polarisation vector will precess around the local magnetic induction of the magnetised regions. In order to align the magnetisation of different ferrite regions, an external magnetic field $H$ is applied. In this case, the angle of rotation of the polarisation vector is a function of the ferromagnetic material present, and because ferrite is the only ferromagnetic material, this will give the fraction of ferrite. Due to local fluctuations of the magnetic induction (in each ferrite grain) there will be a shortening of the polarisation vector. This will lead to an average particle size.

In the past several experiments were carried out on steels C60 (0.60 wt% C) and 57Cr3 (0.75 wt% C) [1]. In this work results on steels with less carbon are presented.

In section 2.1 the general features of the phase transformations studied in this work are given. For the interpretation of the measurements it is necessary to have a good insight in the magnetisation of the steel. In section 2.2 some characteristics of the magnetisation of proeutectoid ferrite and pearlite are given. The neutron depolarisation experiment is explained in section 2.3. Also the formulas for calculating the ferrite fraction and average particle size are given.

In chapter 3 the experimental set-ups are described. Also the sample composition and initial microstructure of the different steel samples is given. The design of the two different furnaces that have been used in the experiments is given. In section 3.3 the effects on the results caused by the two different sample configurations that were used, are discussed.

The results of the neutron depolarisation experiments are described in chapter 4. Experiments have been carried out on C22 (0.22% C), C35 (0.35 %C) and C45 (0.45 %C). On all three samples isochronal measurements have been performed. On sample C35
two other experiments have been carried out. One experiment at a different wavelength, and one experiment with a varying magnetic field.

In chapter 5 the results are discussed. The transformation behaviour of all three samples is compared. As the magnetisation of the sample is important to correctly interpret the measurements, this is discussed separately in section 5.2. The possible wavelength effect on the result of the experiment is discussed in section 5.3.
2.1 Austenite to ferrite and pearlite transformation

The transformation kinetics of austenite (face-centred-cubic) to ferrite (body-centred-cubic) is dependent on carbon diffusion. In figure 2.1 the iron-carbon diagram is given schematically. The $A_3$ line gives the temperature at which the transformation of austenite to proeutectoid ferrite starts. In lowering the temperature the carbon diffuses out of the ferrite and into the austenite. At a certain concentration of carbon in the remaining austenite, $0.80\text{wt}\%\text{C}$, and a maximum concentration of carbon in ferrite, $0.02\text{wt}\%\text{C}$, no longer ferrite is formed, but the pearlite transformation starts. The temperature at which the pearlite transformation starts is called the $A_1$ temperature. Ferrite formed above the $A_1$ temperature is referred to as proeutectoid ferrite.

![Iron-carbon diagram](image)

Figure 2.1 The iron-carbon diagram.

A steel containing less than $0.80\%\text{C}$ (eutectoid composition) is called a hypoeutectoid steel. When a hypoeutectoid steel is allowed to cool slowly (furnace cool) after heating to the austenite phase, the final microstructure of the steel will consist of pro-eutectoid ferrite (containing less than $0.02\%\text{C}$) lining the grain boundaries, since it preferentially nucleates at grain boundaries, and pearlite inside the grains. Pearlite is a mixture of two phases: ferrite and cementite in the form of parallel lamellae. The composition of pearlite is constant: 87.5% ferrite, 12.5% cementite. A region of pearlite with parallel lamellae is called a pearlite colony. In a former austenite grain several pearlite colonies can be formed. The lamellar distance of pearlite is proportional to the undercooling below the $A_1$ temperature. The lamellar structure is not favourable in energy considerations. If the pearlitic structure is held at a relatively high temperature (but below the $A_1$ temperature) globularisation of the cementite lamellae can occur [2].

Proeutectoid ferrite which forms from moderately undercooled austenite (between the $A_1$ and the $A_3$ temperature) has a characteristic morphology. The ferrite nucleates at favourable sites at grain boundaries and grows relatively fast along the grain boundaries, but also into the interior of the austenite grain with a somewhat irregular shape which does
not reflect the internal crystalline structure. This morphology is usually termed as allotriomorphic ferrite. In modelling the ferrite growth, the grains are usually roughly approximated by simple geometric bodies as a sphere, a section of a sphere or an oblate ellipsoid [3].

The transformation of austenite to ferrite can be described by the growth equations of Zener [4], assuming diffusional growth. The radius of a spherical particle, \( \delta \), varies with time according to the equation:

\[
\delta = \alpha_3 \sqrt{D(t - t_n)}, \tag{2.1}
\]

\( \alpha_3 \) is the growth coefficient, \( D \) is the diffusion coefficient of carbon in austenite and \( t_n \) is the time at which the particle starts growing. The growth coefficient depends on the carbon concentrations through:

\[
\alpha_3 = k_i \left( \frac{c^\gamma - c^{eq}_\alpha}{c^\alpha - c^\gamma} \right)^{k_2}, \tag{2.2}
\]

with \( c^\alpha \) and \( c^\gamma \) the equilibrium carbon concentrations in ferrite and austenite, as given by the phase diagram. \( c^\gamma \) is the carbon concentration in the austenite grain, far from the moving boundary. Zener uses the parameters \( k_i = 2.102 \) and \( k_2 = 0.5871 \).

The formula giving the temperature and carbon concentration dependence of the diffusion coefficient \( D^\gamma \) of austenite [5]:

\[
D^\gamma_c = c_1 \left( 1 + y_c (1 - y_c) \frac{c_2}{T} \right) e^{- \frac{(1 - c_3)}{c_4 + c_5 y_c}}, \tag{2.3}
\]

with

\[
y_c^\gamma = \frac{x_c^\gamma}{1 - x_c^\gamma}. \tag{2.4}
\]

\( x_c^\gamma \) is the atom fraction of carbon in austenite. The parameters used are: \( c_1 = 4.53 \cdot 10^{-7} \) m²s⁻¹, \( c_2 = 8339.9 \) K, \( c_3 = 2.221 \cdot 10^{-4} \) K⁻¹, \( c_4 = 17767 \) K, \( c_5 = 26436 \) K.
2.2 Magnetism in steels

To be able to correctly interpret neutron depolarisation experiments, knowledge about the magnetisation of steels is necessary. Iron in the body centred cubic state, whether it is proeutectoid ferrite or ferrite in pearlite, is ferromagnetic below the Curie temperature. Austenite and cementite are paramagnetic in the temperature range of interest. To be able to calculate a ferrite fraction and grain size from the neutron depolarisation experiments the magnetisation must be considered. This magnetisation depends on the microstructure. As the microstructure is constantly changing during the whole transformation (and maybe even after the transformation, if globularisation takes place), it is important to know something about the behaviour of the magnetisation.

In paragraph 2.2.1 an introduction in magnetic domains is given, explaining some terms which are used in this section. In paragraph 2.2.2 the magnetic properties of ferrite and pearlite, and of their combination are described to give some insight in the magnetisation process. In paragraph 2.2.3 the domain size is discussed. Some relationships between domain size and grain size for carbon steels are given. If pearlite is held at a relatively high temperature (but below the A₁ temperature) globularisation can occur. This means that the cementite lamellae will transform into cementite globules, which will act as non-magnetic inclusions. In paragraph 2.2.4 the effect of globular non-magnetic inclusions in steels on the magnetisation is described.

2.2.1 Magnetic domains

In a non magnetised, ferromagnetic material, the atomic magnetic moments are essentially locally directed parallel in domains. Within one domain the magnetisation is close to saturation. A one-domain specimen has a large magnetostatic energy, this is the energy of the magnetic stray field around the domain. A state in which the specimen is divided into several domains (multi domain) lowers this energy strongly, but increases the domain wall energy. Closure domains can lower even more the energy by short-circuiting the domain fluxes within the material. They can be nucleated by defects and by the surface of the specimen. Usually the closure domains are the last domains to reorient or to disappear in the magnetising process.

Multi domain grains can only be formed if the reduction in magnetostatic energy is larger than the energy necessary to form domain walls. The domain wall is a transition area, in which the magnetic moments rotate. The domain wall energy is the difference in energy of the magnetic moments within a domain wall and a moment that is part of a domain. Domain walls can be pinned by impurities, grain boundaries or by magnetic inclusions (these are second phase particles with different magnetic properties than the matrix material). The energy of the domain wall is lowered as soon as the domain wall approaches such a defect. This hinders domain wall movement, and thus the magnetisation process.
In an anisotropic magnetic material, like steel, the magnetic moments will tend to lie along specific crystallographic directions. These directions are called the magnetic easy axes. If a field is applied, first the domains with a direction close to the applied field direction grow. At the cost of these domains, the other domains become smaller. At higher fields rotation of the magnetic moments takes place. The domains which are non-favourably directed overcome the anisotropy energy and align in the crystallographic direction closest to the applied field direction. This is a discontinuous, irreversible rotation of the magnetic moments. At even higher fields, a coherent rotation takes place, all domains are in the direction of the applied field; it seems as if the specimen is a one-domain specimen.

Figure 2.2 Four extreme magnetisation curves, schematically drawn.

In figure 2.2 four extreme magnetisation curves are drawn. If the magnetisation curve at high fields is horizontal this means that there is saturation. Local anisotropy increases the field at which saturation occurs. In the situation of a magnetisation curve with hysteresis, not all particles are perfect spheres. If there is very strong anisotropy then it is possible that at high fields the curve is almost horizontal, but only at very high fields it will really be horizontal.

Figure 2.3 Hysteresis loop of a polycrystalline ferromagnetic material.
In figure 2.3 a hysteresis loop of a polycrystalline ferromagnetic material is given. In this curve the magnetisation $M$ is given as a function of the applied external magnetic field $H$. $M_s$ is the saturation magnetisation. If the sample is saturated, all magnetic dipoles in the material are oriented in the same direction, namely in the direction of $H$.

$$M_s = n p,$$  \hspace{1cm} (2.5)

$n$ is the number of elementary atomic magnetic dipoles per unit volume, each with magnetic moment $p$. $M_r$ is the remanent magnetisation, the magnetisation which remains after the field has been removed. $H_c$ is the coercive field, the magnetic field necessary to reduce the magnetisation to 0. If $H_c$ is smaller than $1000 \text{ A/m}$ the material is said to be magnetically soft. If $H_c$ is larger than $10,000 \text{ A/m}$ it is a magnetically hard material. More or stronger pinning sites will increase the coercivity, because the domain walls will have more difficulty in moving through the material. If a magnetic field $H$ is applied, the medium will react with a magnetic induction $B$.

2.2.2 Domains in proeutectoid ferrite and pearlite

In proeutectoid ferrite, the domain walls are mostly pinned by grain boundaries, because there is a change in crystal orientation at these grain boundaries. The maximum distance a domain wall can cross before it will get pinned is called the maximum domain wall jump. In proeutectoid ferrite the maximum domain wall jump depends on grain size [6].

In an eutectoid steel (0.80\% carbon) you can get a proeutectoid ferrite film on the former austenite grain boundaries, with one to twenty pearlite colonies inside. In figure 2.4 a schematically view of this situation is given. The proeutectoid ferrite film isolates the pearlite colonies, so the colonies inside one former austenite grain have no orientation relations to the colonies in other former austenite grains. The size of the volume surrounded by the proeutectoid ferrite film defines the maximum domain wall jump [6].

![Figure 2.4: Proeutectoid ferrite film on the former austenite grain boundary, isolating several pearlite colonies.](image)

Despite the differences in microstructure, the domain walls in pearlite colonies have about the same dimensions and distributions as the domain walls in proeutectoid ferrite grains [6]. A domain wall could easily move through a uniform, geometrical perfect lamellar structure. In the case of pearlite there are bended lamellae and parts with disordered
lamellae, leading to variations in the magnetostatic energy of the domain wall, depending on its position [7]. An irregular pearlite structure gives an irregular domain pattern. If the pearlite structure is quite regular, extra pinning arises at the boundary between two pearlite colonies [8]. It is more difficult for domain walls to pass through pearlite colonies than through ferrite grains, because pearlite has a more complex magnetic structure [9,10]. As a result, the coercivity in pearlite will be higher than in proeutectoid ferrite.

Domain walls in pearlite are often found [11] to be parallel to the lamellae, but sometimes walls are found which cross lamellae. Fine lamellar distances in pearlite exclude the possibility of creating closure domains within one lamella. Domains found experimentally in pearlite are larger than the lamellar distance (see figure 2.5) in all directions [7]. Below the Curie temperature of cementite, domain walls are mostly found inside cementite lamella, because the domain wall energy inside cementite is lower than inside ferrite. But because the domain wall energy is also dependent on the orientation of the magnetisation with respect to the crystallographic and lamellar directions, domain walls are also found in ferrite lamellae. In the temperature range of interest the cementite is paramagnetic, so domain walls can only exist in the ferritic phase of the pearlite. It is most probable that different parts of domain wall over several ferrite lamellae will behave like they are one domain wall. In figure 2.5 this situation is schematically drawn.

![Figure 2.5](image)

Different domain walls in different ferrite lamellae, moving like they are one domain wall.

Cementite is only a strong pinning site if the domain walls are directed parallel to the lamellae. If, at a certain magnetisation, the majority of the moving domain walls are walls which make high angles to the cementite lamellae, the cementite lamellae are no longer strong pinning sites [6]. Increasing the carbon content increases the density of pinning sites [12], and therefore will increase the coercivity. If the grain size decreases, the average mean free path between the grain boundaries decreases. As the grain boundaries form pinning sites, the density of pinning sites will increase and therefore the coercivity will increase. In carbon steels with more than 0.2%C, the grain size effect is negligible compared to the carbide effect, as will be described in paragraph 2.2.4.

If there is a ferrite/pearlite boundary, the domain walls in the ferrite grains are often pinned to this boundary. If an external field is applied, the pinned domain wall will bow. At high
magnetisations the ferrite/pearlite boundary is a better pinning site than the ferrite/ferrite or pearlite/pearlite boundary [10].

If a domain wall is perpendicular to a cementite lamella, and the wall is between a domain with magnetisation parallel to the applied field and a domain with magnetisation anti parallel to the applied field, then the wall can move easily. (See figure 2.6) A problem arises if the wall approaches at another colony, with lamellae in different directions. Only if a more complex domain structure is formed will the wall still be able to move [10].

2.2.3 Domain (wall) size

The domain wall thickness is determined by the competing influences of two factors: the anisotropy energy and the exchange energy. The anisotropy energy is proportional to the number of layers of atoms in the domain wall and of the lattice spacing, with a spin orientation not in an easy direction. This energy tends to make the domain walls thinner because the anisotropy energy is lowest with all moments aligned along crystallographically equal axes. The exchange energy arises because of interactions between the magnetic moments and is short ranged limited to nearest neighbours. This energy tends to make walls thicker since the exchange energy in a ferromagnet is minimised when neighbouring magnetic moments are aligned parallel.

The domain wall thickness is determined by the energy minimisation of these two energies. The domain wall thickness \( \delta_w \) can generally be written as:

\[
\delta_w = \frac{J}{\sqrt{K}},
\]

where \( J \) is the exchange energy and \( K \) is the anisotropy energy.

The largest domains are likely to occur on grains bound by (100) faces and the smallest on grains with (111) faces, since the energies of the closing structures at these faces have minimum and maximum values respectively.
Experimentally [12] for carbon steels the relations between ferrite grain size and domain size have been determined. For grains bound only by (100) faces, the maximum mean domain width $D_{\text{max}}$ is given in terms of the mean length $L$ in the (100) direction by:

$$D_{\text{max}} = 2\sqrt{\frac{E}{c_{11}|L|^2}}, \quad (2.7)$$

$c_{11}$ is an elastic constant, $E$ is the energy of a 180° Bloch wall and $l$ is the magnetostrictive constant.

The minimum domain width, $D_{\text{min}}$, is given by:

$$D_{\text{min}} = 0.7\left(\frac{E}{K}\right)^{\frac{1}{3}} L^2, \quad (2.8)$$

in which $K$ is the anisotropy constant.

Bloor [13] calculated the domain widths for 3% Si-Fe using: $c_{11} = 2.2 \cdot 10^{11}$ J m$^{-3}$, $E = 1.4 \cdot 10^{-3}$ J m$^{-2}$, $l = 2.7 \cdot 10^{-5}$, $K = 3.6 \cdot 10^{7}$ J m$^{-1}$. The values of these constants do not differ greatly form those of slightly impure iron.

Their calculation showed that there is an obvious tendency for the experimentally measured domain widths to approach formula (2.7) for the larger grains and formula (2.8) for small grains. The density of domain walls is proportional to $\frac{1}{D}$. Thus, the density of domain walls would in general decrease with the increase in grain size.

### 2.2.4 Globules

If, at some point in the transformation, globularisation of the cementite takes place, there will be a totally different magnetic behaviour. The non magnetic globular cementite has free poles and therefore a large magnetostatic energy, as can be seen in figure 2.7.

![Figure 2.7 A spherical non-magnetic inclusion.](image)

As soon as a domain wall tries to pass through the inclusion, the free poles will change, and this will reduce the magnetostatic energy (See figure 2.8). As this is a situation of lowest energy, the domain wall will be pinned by the inclusion [14].
Reality, however, is somewhat more complex. In the case of a cubic anisotropy spike domains will form around an inclusion, which leads to a further reduction in magnetostatic energy. In figure 2.9 two different situations with spike domains around a non-magnetic inclusion are drawn.

Cementite in ferrite is a non magnetic globule in a cubic anisotropy. Therefore these spike domains can provide connections to the other cementite globules. A lamellar structure will give more pinning than a structure with globules. Globularisation of a certain steel will decrease the coercive force [7].
2.3 Neutron depolarisation

In order to be able to study the ferrite fraction and particle size during the austenite/ferrite and austenite/pearlite transformation, the neutron depolarisation technique is used. In paragraph 2.3.1 a neutron depolarisation experiment is described, and an insight is given in the processing of the data. All the formulas given are for a field applied in the y-direction, as was used in the experiments. The ferrite fraction and the particle size are determined from the difference in polarisation vector before and after the neutron beam has passed through the sample. In paragraph 2.3.2 the formulas for the calculation of ferrite fraction and particle size are given.

2.3.1 Neutron depolarisation measurement

In a neutron depolarisation experiment, both the mean magnetic induction and the local fluctuations of the magnetic induction are measured. Below the Curie temperature, the transformation of austenite to proeutectoid ferrite is the formation of ferromagnetic ferrite, in a matrix of paramagnetic austenite. The transformation of austenite to pearlite is the formation of lamellae of ferromagnetic ferrite and paramagnetic cementite, in the temperature region of interest (the Curie temperature of cementite is 210 °C [15]). Over the entire experimental range, magnetisation only takes place in the ferrite. The amount of ferrite can be determined from the mean magnetic induction. Simultaneously the correlation parameter of ferrite in austenite, or the correlation parameter of pearlite, can be determined from the local fluctuation of the magnetic induction.

Neutrons possess a spin (with spin vector S). If the average spin component of a neutron beam is not zero the beam is polarised. The polarisation is given by:

\[
P = \frac{I_+ - I_-}{I_+ + I_-} = <\mathbf{\sigma}>.
\]  

(2.9)

where \(I_+\) is the measured spin up intensity, with spin parallel to \(<\mathbf{\sigma}>\). \(I_-\) is the measured intensity, with spin antiparallel to \(<\mathbf{\sigma}>\). The direction of \(\mathbf{P}\) is that direction in which \(P\) is a maximum:

\[
\mathbf{P} = <\mathbf{\sigma}>.
\]  

(2.10)

The polarisation vector \(\mathbf{P}\) of the neutron beam will rotate around any magnetic field that is passed during transmission, which is described by the Larmor equation [16]:

\[
\frac{d\mathbf{P}}{dt} = \gamma [\mathbf{P}(t) \times \mathbf{B}(t)],
\]  

(2.11)

in which \(\gamma\) is the gyromagnetic ratio of \(1.83 \times 10^8 \text{s}^{-1}\text{T}^{-1}\) and \(\mathbf{B}\) is the local magnetic induction.

The local magnetic induction in a sample can be described as being composed of two parts:

\[
\mathbf{B}(r) = <\mathbf{\bar{B}}> + \Delta \mathbf{B}(r).
\]  

(2.12)
The mean magnetic induction in the sample \( \langle \vec{B} \rangle \) results in a rotation of the total polarisation vector over an angle \( \phi \). The local fluctuations of the magnetic induction \( \Delta \vec{B}(r) \) result in a shortening of the polarisation vector by an amount proportional to the correlation parameter \( \xi \) of the fluctuation. In figure 2.10 the rotation and shortening of the initial polarisation vector \( \vec{P}^0 \) is sketched.

![Figure 2.10](image)

Figure 2.10  Rotation and depolarisation of the initial polarisation \( \vec{P}^0 \) around \( \vec{B} \).

The relationship between the mean magnetic induction and the rotation angle can be written as:

\[
\phi = \langle \vec{B} \rangle L c, \quad (2.13)
\]

\( L \) is the sample thickness, \( c = \frac{\gamma^2}{v^2} = 2.18 \times 10^{20} \text{[T}^{-2} \text{m}^{-4}] \lambda^2 \), where \( v \) is the velocity and \( \lambda \) the wavelength of the neutron beam.

The mean polarisation vector \( \vec{P} \) after transmission through a sample can be related to the incoming mean polarisation vector \( \vec{P}^0 \) by:

\[
\vec{P} = \hat{D} \vec{P}^0, \quad (2.14)
\]

where \( \hat{D} \) is the (3x3) depolarisation matrix. The elements \( D_{ij} \) of the depolarisation matrix are determined from intensity measurements.

\[
D_{ij} = \frac{1 - I_j}{I_x}, \quad (2.15)
\]

where \( I_x \) is the intensity of the completely depolarised neutron beam and \( I_j \) is the intensity measured with polarisation in direction \( j \) of the laboratory system, and the analysis in direction \( i \) of the laboratory system. \( Q_0 \) is the polarising power of the polariser/analyser combination. The depolarisation is measured in all nine possible combinations of polarisation- and analysis directions.

In the case of a mean magnetic induction in the \( y \)-direction, the precession angle \( \phi \) can directly be calculated from the measured depolarisation matrix via:
The precession of the polarisation around a magnetic induction $\vec{B}$ with direction indices $(n_x, n_y, n_z)$ can be described using the rotation matrix $\hat{R}$:

$$\hat{R}(\varphi) = \hat{U} + \hat{S} \sin \varphi + \hat{S}^2 (1 - \cos \varphi),$$

where $\hat{U}$ is the unit matrix and $\hat{S}$ is given by:

$$\hat{S} = \begin{pmatrix} 0 & -n_z & n_y \\ n_z & 0 & -n_x \\ -n_y & n_x & 0 \end{pmatrix}.$$  

While during the rotation also depolarisation occurs, each sample is divided into identical units along the neutron beam direction.

$$L_w = \frac{L_{\text{z}}}{N},$$

where $L_w$ is the transmission length of the unit. In each unit the rotation angle is $\frac{\varphi}{N}$, this should be representative for the whole sample. The magnetic induction in each unit is assumed to be equal to the mean magnetic induction $\langle B \rangle$.

The depolarisation matrix after a transmission length $L_w$ through the sample is given by:

$$D'_{ij} = \delta_{ij} (1 - c_{L_w} \xi) + c_{L_w} \alpha_{ij},$$

$\delta_{ij}$ is the Kronecker delta, $\alpha_{ij}$ are the elements of the correlation matrix and $\xi$ the correlation parameter. The correlation matrix is given by:

$$\alpha_{ij} = \frac{1}{W_w} \int d^3r \int_{x_0} dx' \Delta B_i(x) \Delta B_j(x'),$$

$W$ is a small representative volume of the medium which lies between $x_0$ and $x_0 + L_w$. $x$ is the propagation direction of the neutron beam through the sample. The correlation parameter can be calculated from the correlation matrix:

$$\xi = \sum \alpha_{ii}.$$  

If there are no correlations between $\Delta B_i(r)$ and $\Delta B_j(r)$ for $i \neq j$, then all non-diagonal elements of $\hat{D}'$ are 0, and the depolarisation matrix $\hat{D}'$ for the whole sample is given by:

$$\hat{D}' = \begin{bmatrix} 1-c_{L_w}(\xi-\alpha_{xx}) & 0 & 0 \\ 0 & 1-c_{L_w}(\xi-\alpha_{yy}) & 0 \\ 0 & 0 & 1-c_{L_w}(\xi-\alpha_{zz}) \end{bmatrix}.$$  

The total interaction of the polarisation vector can be described by:

$$\hat{D} = \left( \hat{R} \left( \frac{L_w}{2} \right) \hat{D}' \hat{R} \left( \frac{L_w}{2} \right) \right)^N.$$
The correlation parameter \( \xi \) and \( \alpha_{yy} \) can be directly calculated from the depolarisation matrix via:

\[
\xi = \frac{-\ln(\det(\hat{D}))}{2cL_s},
\]

(2.25)

\[
\alpha_{yy} = \frac{\ln(D_{yy}) - \frac{1}{2}\ln(\det(\hat{D}))}{cL_s}.
\]

(2.26)

The mean local orientation of \( \Delta B(r) \) is expressed in the quantity \( \gamma \):

\[
\gamma = \frac{\langle \Delta B^2 \rangle}{\xi} = \frac{\langle \Delta B^2 \rangle}{<B^2>},
\]

(2.27)

\( \gamma \) is the average of the normalised induction variation projection on the y-axis.

The polarising power of the polariser (P) and analyser (Q) is not 100%, and is wavelength dependent. At a temperature above the Curie temperature a calibration procedure is performed which corrects for these effects.

In the data processing program more corrections are performed [16]. The formulas given here only give some insight into the relationship between the measured depolarisation matrix \( \hat{D} \) and parameters of interest \( \varphi, \alpha, \) and \( \xi \). Equations (2.16, 2.25, 2.26) are only valid for a situation where there is no magnetic field in the x- and z-direction, only in the y-direction.

2.3.2 Calculation of the ferrite fraction and particle size

The mean magnetic induction \( <B> \) of the sample with paramagnetic and ferromagnetic material can be written as:

\[
<B> = f m B_s(T),
\]

(2.28)

where \( f \) is the fraction ferromagnetic material and \( m \) is the magnetisation factor:

\[
m = \frac{<n_y>}{B_s} = \frac{\overline{B}}{B_s},
\]

(2.29)

where \( \overline{B} \) is the magnetic induction in the direction of the applied field, averaged over all magnetic domains. The temperature dependence of the spontaneous magnetic induction \( B_s \) used is taken from experimental data measured on pure iron by Stüsser [17]:

\[
B_s(T) = B_0 \left( \frac{T_c - T}{T_c} \right)^{0.35} \left[ 1 - 0.5 \left( \frac{T_c - T}{T_c} \right)^{0.35} \right],
\]

(2.30)

\( B_0 \) is 3.454 [T], \( T \) and \( T_c \) are given in Kelvin. In figure 2.11 the spontaneous magnetic induction is plotted as a function of the temperature for a Curie temperature of 773 °C.
The spontaneous magnetic induction for pure iron versus temperature, using $T_c=1046$ K.

The magnetic volume fraction is:

$$f = \frac{\varphi}{mB_L\sqrt{c}}.$$  \hspace{1cm} (2.31)

If it is assumed that all domains are spheres, homogeneously distributed, with no overlap of magnetic fields of the different domains, then one can derive the particle size. One also has to know whether each particle consists of one or more domains. Here it is assumed that each proeutectoid ferrite particle is a single domain. The particle size can be calculated from the correlation parameter $\xi$ by taken into account the mean magnetisation of the sample. If the magnetisation is large the local fluctuation of the magnetisation will be small. The average particle radius is now given by:

$$\delta = \frac{3\xi c_3}{2fB_L^2(1-c_2c_3m^2)},$$ \hspace{1cm} (2.32)

with

$$c_2 = \left(\frac{4\pi f^2}{81}\right)^{1/3}$$ \hspace{1cm} (2.33)

and

$$c_3 = \left(\frac{1}{3-<n_y^2>}\right)^{1/3}$$ \hspace{1cm} (2.34)

where $<n_y^2>$ is the average of the square of the $y$-component of the magnetisation.

The average particle size measured by neutron depolarisation is related to the average particle radius $r$ by:

$$\delta = \frac{<r^4>}{<r^3>},$$ \hspace{1cm} (2.35)
The quantity $\gamma_f$ of the sample, as defined in equation (2.27) equals:

$$\gamma_y = \frac{\gamma_y^0 - c_2 c_3 m_y}{1 - c_2 c_3 m^2},$$

with

$$\gamma_y^0 = \frac{0.25 + 1.25 \langle n_y^2 \rangle}{3 - \langle n_y^2 \rangle}.$$  

(2.36)

(2.37)

The rotation angle $\varphi$ and the correlation parameter $\xi$ are obtained from the measurement. To be able to calculate the ferrite fraction $f$ and the particle size $\delta$ values for $m$ or $<n_y^2>$ are necessary. In our case, to process the experimental data a constant mean magnetisation, $m$ is chosen when a constant field is applied during the experiments.
3 Experimental

3.1 Sample characterisation

3.1.1 Composition and initial microstructure

Three different steel compositions have been studied: C22, C35 and C45. They are normal industrial steels for which the steel compositions are given in table 3.1. The analysis was carried out at Hoogovens. The carbon analysis has been done by gas analysis on a Ströhlein CS-mat and the other elements have been analysed by X-ray Fluorescence Spectrometry. The most important elements in the studied steels are carbon and manganese. Increasing the manganese concentration decreases the Curie temperature. The austenisation temperatures used are not high enough to redistribute the manganese homogeneously. Therefore if there is any texture apparent in the sample with a non-homogeneous distribution of manganese, it will stay the same after the transformation.

Table 3.1 Steel composition (±0.005 wt%).

<table>
<thead>
<tr>
<th></th>
<th>C (wt%)</th>
<th>Mn (wt%)</th>
<th>S (wt%)</th>
<th>Si (wt%)</th>
<th>P (wt%)</th>
<th>Cr (wt%)</th>
<th>Ni (wt%)</th>
<th>Cu (wt%)</th>
<th>Mo (wt%)</th>
<th>Sn (wt%)</th>
<th>N (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C22</td>
<td>0.214</td>
<td>0.513</td>
<td>0.031</td>
<td>0.2</td>
<td>0.019</td>
<td>0.021</td>
<td>0.049</td>
<td>0.086</td>
<td>0.003</td>
<td>0.003</td>
<td>0.0071</td>
</tr>
<tr>
<td>C35</td>
<td>0.364</td>
<td>0.656</td>
<td>0.021</td>
<td>0.305</td>
<td>0.014</td>
<td>0.177</td>
<td>0.092</td>
<td>0.226</td>
<td>0.016</td>
<td>0.017</td>
<td>0.00106</td>
</tr>
<tr>
<td>C45</td>
<td>0.468</td>
<td>0.715</td>
<td>0.031</td>
<td>0.257</td>
<td>0.002</td>
<td>0.193</td>
<td>0.144</td>
<td>0.231</td>
<td>0.017</td>
<td>0.013</td>
<td>0.0091</td>
</tr>
</tbody>
</table>

Figure 3.1 Initial microstructure of C22, 200x. The light areas are proeutectoid ferrite grains, the dark areas are pearlite colonies.
Figure 3.2  Initial microstructure of C35, 200x. The light areas are proeutectoid ferrite grains, the dark areas are pearlite colonies.

Figure 3.3  Initial microstructure of C45, 200x. The light areas are proeutectoid ferrite grains, the dark areas are pearlite colonies.
In the figures 3.1 to 3.3 the initial microstructure is given. This consists of a ferrite matrix with pearlite colonies. The sizes of the ferrite grains and pearlite colonies are given in table 3.2.

The initial microstructure of C35 shows a rolling texture in the direction perpendicular to the picture of figure 3.2, C22 and C45 do not have any rolling texture.

Table 3.2 Grain diameters of the initial microstructures.

<table>
<thead>
<tr>
<th></th>
<th>ferrite grain diameter (µm)</th>
<th>pearlite colony diameter (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C22</td>
<td>30</td>
<td>14</td>
</tr>
<tr>
<td>C35</td>
<td>18</td>
<td>20</td>
</tr>
<tr>
<td>C45</td>
<td>17</td>
<td>33</td>
</tr>
</tbody>
</table>

3.1.2 Thermodynamical calculations

On base of the steel compositions some thermodynamical calculations have been carried out. The program MTDATA is a thermodynamical database, with which one can calculate the transformation temperatures and fractions proceeding on local equilibrium. The $A_1$ upper and lower temperature, the $A_3$ temperature and the Curie temperature have been calculated. To determine the Curie temperature the composition of the proeutectoid ferrite according to local equilibrium has been used. The Curie temperature depends mainly on the manganese concentration. In table 3.3 the calculated values are given.

Table 3.3 Calculated $A_1$, $A_3$ and $T_c$ temperatures.

<table>
<thead>
<tr>
<th></th>
<th>$A_1$ upper (°C)</th>
<th>$A_1$ lower (°C)</th>
<th>$A_3$ (°C)</th>
<th>$T_c$ (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C22</td>
<td>717</td>
<td>701</td>
<td>823</td>
<td>761</td>
</tr>
<tr>
<td>C35</td>
<td>721</td>
<td>692</td>
<td>782</td>
<td>758</td>
</tr>
<tr>
<td>C45</td>
<td>722</td>
<td>693</td>
<td>760</td>
<td>759</td>
</tr>
</tbody>
</table>

Thermocalc is a similar program as MTDATA. With this program the equilibrium ferrite fraction as a function of temperature has been calculated. In figure 3.4 the results are given for the three steel compositions. From this figure the total ferrite fraction (proeutectoid ferrite and ferrite in pearlite) and the maximum proeutectoid ferrite fraction can be derived. These are given in table 3.4.

Table 3.4 Calculated ferrite fractions.

<table>
<thead>
<tr>
<th></th>
<th>maximum proeutectoid ferrite fraction</th>
<th>total ferrite fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>C22</td>
<td>0.71</td>
<td>0.96</td>
</tr>
<tr>
<td>C35</td>
<td>0.47</td>
<td>0.93</td>
</tr>
<tr>
<td>C45</td>
<td>0.31</td>
<td>0.91</td>
</tr>
</tbody>
</table>
3.1.3 DTA measurements

Differential Thermal Analysis experiments \[17\] were carried out to measure the Curie temperature. In DTA experiments the difference in temperature (\(\Delta T\)) between a reference and the sample is measured. Heat effects at the different transformations appear as a peak. As there is also a heat effect at the magnetic transition, the Curie temperature can also be measured. There will only be a magnetic transition if ferrite is present, so only if the \(A_3\) temperature is higher than \(T_c\). For the three steel compositions of this work, only the Curie temperature of C22 could be estimated by DTA measurements.

In the experiments performed, the DTA apparatus was calibrated for a cooling rate of 20 °C/min. Because of a delay time between the thermocouple signal and the apparatus, there might be a shift in the DTA results to lower (cooling) and higher (heating) temperatures. The Curie temperature should be independent of cooling rate and should be the same in a cooling and a heating course \[18\].

In figure 3.5 the results of the DTA measurement on C22 are given. As the apparatus was calibrated for a cooling rate of 20 °C/min, only the curves for this cooling rate have been used. The heating curve shows a slightly lower Curie temperature than the cooling curve. In view of the above mentioned, this result seems strange.
Figure 3.5  DTA measurement for C22. 1: cooling at 20 °C/min, 2: cooling at 5 °C/min, 3: heating at 5 °C/min, 4: heating at 20 °C/min. At about 760 °C in all curves a little peak is visible, this will be a peak due to the magnetic transition at the Curie temperature.

The large peaks in figure 3.5 arise from the austenite to pearlite or vice versa transformation. As these are measured at much larger cooling and heating rates than used in the neutron depolarisation measurements these cannot be compared to the results of the transformation experiments in chapter 4.

Figure 3.6  DTA measurement of C22. Enlargement from figure 3.5 of the curve for a cooling rate of 20°C/min.
In figure 3.6 the peak resulting from the magnetic transition in the cooling curve of 20 °C/min is enlarged and a Curie temperature of 756 °C can be derived from this curve.

Also DTA measurements have been performed on relatively pure iron. The exact composition is given in table 3.5.

Table 3.5 Iron composition

<table>
<thead>
<tr>
<th></th>
<th>Fe</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cl</th>
<th>K</th>
<th>Ca</th>
<th>Ta</th>
</tr>
</thead>
<tbody>
<tr>
<td>iron</td>
<td>99.87±0.04</td>
<td>0.05±0.02</td>
<td>0.0067±0.0007</td>
<td>0.004±0.001</td>
<td>0.010±0.003</td>
<td>0.009±0.004</td>
<td>0.0084±0.0009</td>
<td>0.013±0.006</td>
</tr>
</tbody>
</table>

The Curie temperature of pure iron is about 773 °C. In figure 3.7 the results of the DTA measurement on iron are given. The Curie temperature derived from the cooling curve (number 5) is 764 °C. The Curie temperature derived from the heating curve, number 6, seems a bit higher.

![Figure 3.7](image)

**Figure 3.7** DTA measurement on iron. 1: cooling at 20 °C/min, 2: heating at 20 °C/min, 3: cooling baseline, 4: heating baseline, 5: corrected cooling curve (1-3), 6: corrected heating curve (2-4).

As the results of the DTA measurements are not very trustworthy, they are not used in processing the data from the neutron depolarisation experiments.

3.1.4 Austenite grain diameters

Salt bath experiments were performed to determine the austenite grain diameter: the samples were austenitised at the same temperatures as in the neutron depolarisation experiments. After austenitisation they were quenched in water, and the austenite grain
diameter was determined from the resulting microstructure. These grain diameters are given in table 3.6.

Table 3.6 Austenite grain diameter.

<table>
<thead>
<tr>
<th></th>
<th>austenite grain diameter (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C22</td>
<td>19.0 ± 2.5</td>
</tr>
<tr>
<td>C35</td>
<td>13.6 ± 2.5</td>
</tr>
<tr>
<td>C45</td>
<td>18.9 ± 2.5</td>
</tr>
</tbody>
</table>

3.2 Neutron Depolarisation set-up

Measurements have been performed on the crystal polarimeter (KP) and the mirror polarimeter (SP).

In the crystal polarimeter the white neutron beam is monochromatised by Bragg reflection from a pyrolytic graphite crystal and polarised to 98% by reflection on a stack of polarising mirrors. This gives a neutron beam with a wavelength of 0.16 ± 0.00316 nm. By means of polarisation rotators in front of the sample, the direction of the polarisation vector can be selected along the x-, y- or z-axis. Polarisation rotators behind the sample allow 3-dimensional polarisation analysis of the transmitted beam. In figure 3.8 a schematic drawing of the set up is given.

![Schematic drawing of the set-up crystal polarimeter.](image)

The set-up of the mirror polarimeter is more or less the same as the crystal polarimeter, only here the white beam passes through the sample, and after passing the analyser, the beam can be reflected by a crystal to produce different wavelengths at different Bragg angles. There are 31 detectors, so measurements of 31 different wavelengths can be done. In the experiments performed on the mirror polarimeter the beam was reflected by a pyrolytic graphite crystal. Only one detector was used. For this specific detector the intensity as a function of wavelength is given in figure 3.9. A rather broad spectrum at a wavelength of 0.123 nm could be obtained.
Figure 3.9  Intensity versus wavelength for the detector used in the mirror polarimeter experiments.

3.3 Sample configuration

In the neutron depolarisation experiments two different furnaces, with two different sample shapes have been used. In figure 3.10 a schematic view of a ring sample with a banana-shaped diaphragm is given. In this configuration there is only one coil of molybdenum wire around the sample, with 4 windings per centimetre. On the KP only these ring-shaped samples have been measured.

![Diagram of a ring sample with a single coil](image)

Figure 3.10  Sample environment for ring-shaped sample with a single coil.

In figure 3.11 a schematic view is given of a rectangular sample, with a rectangular diaphragm. In this configuration there are four coils of molybdenum wire placed above one another, each having 8 windings per centimetre. Together this configuration gives 32 windings per centimetre. Therefore, much higher fields can be obtained. A boron nitride
plate contains the molybdenum wire. This plate is positioned in an iron ring, which provides flux closure. The rectangular sample has been used in the SP experiment.

**Figure 3.11** Sample environment for the rectangular sample with four coils.

In figure 3.12a and b a side-view and a top view of this sample configuration is given. On both sides of the sample there is an iron ring, in which the boron nitride is placed.

**Figure 3.12** Side view (a) and top view (b) of the rectangular sample environment, with four layers of molybdenum coils.

Sample C35 has a very clear rolling texture which will probably have an effect on the magnetisation, especially in the rectangular sample. In figure 3.13 and 3.14 the sample shape and the direction of the rolling texture are drawn for both situations. In the rectangular sample homogeneous ferrite layers can be found in the direction of the applied field, so this will be much easier to magnetise than the ring-formed sample.
direction of the rolling texture

Figure 3.13 Direction of the rolling texture for the ring-formed sample.

The temperature of the sample is measured by a thermocouple, which is placed on the sample, between the molybdenum coils. The error in the measured temperature will be about 3 to 5 °C. In the sample configuration with the ring-shaped sample the molybdenum coils are shielded by ceramic tubes, in the other configuration this is not the case. Therefore the temperature measured on the rectangular sample configuration could deviate from the actual temperature of the sample.

Figure 3.14 Direction of the rolling texture for the rectangular sample.

The temperature of the sample is measured by a thermocouple, which is placed on the sample, between the molybdenum coils. The error in the measured temperature will be about 3 to 5 °C. In the sample configuration with the ring-shaped sample the molybdenum coils are shielded by ceramic tubes, in the other configuration this is not the case. Therefore the temperature measured on the rectangular sample configuration could deviate from the actual temperature of the sample.

Figure 3.15 Heat contribution to the sample comes from the coax and the magnetic windings.

In figure 3.15 the sample configuration of the rectangular sample is given. The sample is heated by the thermocoax heating element outside the cylinder in which the sample is placed. In addition to this, there is also a heat input from the molybdenum coils. At the diaphragm the sample can radiate heat. There are radiation shields around the iron rings, but as the sample is positioned in the centre there is a distance of a few centimetres from the sample to the radiation shields. The radiation shields are therefore more effective for the iron rings than for the sample, with the consequence that the temperature of the sample is lower than the temperature of the iron ring. As the Curie temperature of the ring
is about 770 °C, there will be no flux closure before the iron ring is below this temperature. This implies a complication in the correction procedure for the measurement. Normally, the effect of the applied field in the oven is determined at temperatures above $T_c$ for the sample, but in this configuration such a measurement does not give a useful result, since the field changes strongly at $T=T_c$ for the rings, which practically coincides with $T_c$ for the sample. If the rings to provide flux closure were made of a material with a higher Curie temperature than iron a much better experiment could have been performed.

3.4 Magnetic field

Besides experiments with a constant applied magnetic field, sweep-field experiments have been carried out on both sample shapes. This was done to obtain more information about the magnetic behaviour of the sample at different stages of the transformation. In these experiments the applied external magnetic field changes periodically with time. During each period which lasts a second the intensities are measured in 50 channels. Every channel corresponds to a different value of the applied field. In about 60 seconds one matrix element is measured. To obtain intensities per matrix element in each channel the intensities are summed over the 60 periods. This will give 50 intensities per matrix element. In these experiments a saw-tooth like applied field was used. In figures 3.16 the field of one period as a function of channel number is given.

Figure 3.16  Saw-tooth like field as a function of channel number for the ring shaped sample with 4 windings per centimetre experiment.

From the results of the sweep-field measurements information can be obtained about the magnetic situation of the sample, at each stage of the transformation. Hysteresis loops at different stages can be calculated. Because in each matrix the rotation is also a function of the applied field and therefore of the channel number, the sharp transition in the rotation at the pearlite transformation can be better processed.
3.5 Microstructure

For microstructural examination a part at the diaphragm and a cross-section from each sample is prepared after each experiment. The samples were polished and etched in Nital 2%, for 20 seconds.

To determine the ferrite fraction at the end of the transformation, a CBA 8000 and a Quantimet 570 have been used. By performing quantitative image analysis, the fraction proeutectoid ferrite can be determined from the samples after the transformation.
4 Results

In this chapter the results of the neutron depolarisation experiments on the three different steel compositions are given. In section 4.1 the result of the isochronal experiment with constant magnetic field of sample C45 is given. Similar experiments have been carried out on C35 and C22, for which the results are presented in 4.2 and 4.3.

Further, two additional experiments were carried out on C35 samples. In 4.2.2 the results of the sweepfield experiment is given. Hysteresis curves at different stages of the transformation are given. In section 4.2.3 the effect of the wavelength of the neutrons used to measure the depolarisation matrix is described. A similar isochronal experiment as is described in section 4.2.1 was carried out, but at a smaller wavelength.

![Figure 4.1](image_url) 

**Figure 4.1  Temperature versus time for the isochronal experiments.**

In the isochronal experiments the samples were austenitised and later cooled down with about 10°C/min. At 685 °C (after most of the pearlite transformation) the samples were cooled faster. In figure 4.1 the time-temperature history is given.

4.1 Isochronal transformation experiments on 0.45% C steel (C45)

During this experiment sample 45-2 was held at an austenisation temperature of 850 ºC for one hour and 45 minutes and then cooled according to the temperature course given in figure 4.1. The applied external field was constant 680 A/m. This experiment has been carried out on the crystalpolarimeter, with the sample environment as given in figure 3.10. The sample thickness was 0.280 mm.

In figure 4.2 the rotation as a function of temperature is given for the heating and successive cooling curve of the experiment. The first increase in rotation upon cooling is due to the formation of proeutectoid ferrite. Below 700 ºC a very rapid increase in rotation can be seen, which is due to the transformation of austenite to pearlite. At the start of the transformation during cooling, at approximately 757 °C, no jump in the rotation is visible, which indicates that the formation of proeutectoid ferrite has not yet
started before the ferromagnetic transition. In figure 4.3 the heating and cooling curve of the determinant of the depolarisation matrix is given as a function of temperature. Because the determinant depends on $B^2$, the starting temperature of the measured transformation can be determined at the point where the determinant becomes non-zero. The temperature at which the rotation first starts to deviate from the rotation above the Curie temperature, $T_\phi$, is higher in the heating experiment than in the cooling experiment. $T_\phi$ in the heating experiment will be the Curie temperature or the temperature at which the transformation ends. Because of hysteresis in the start and end temperature of the transformation, $T_\phi$ will be the start transformation temperature. The heating curve is measured to have two values of $T_\phi$, if these are the same, then $T_\phi = T_c$.

The transformed samples were examined by quantitative image analysis to define the proeutectoid ferrite fraction and the total ferrite fraction. To calculate the total ferrite fraction the pearlite composition of 87.5% ferrite and 12.5% cementite was used. In table 4.1 the results are given.

Table 4.1 Fractions derived from the CBA measurement on the transformed C45 sample.

<table>
<thead>
<tr>
<th></th>
<th>fraction proeutectoid ferrite</th>
<th>total ferrite fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>45-2</td>
<td>0.16</td>
<td>0.90</td>
</tr>
</tbody>
</table>

Figure 4.2 Rotation as a function of temperature.

Figure 4.3 Logarithm of the determinant of the depolarisation matrix as a function of temperature.
In figure 4.4 the ferrite fraction, as calculated from the cooling experiment, as a function of temperature is given. The fraction was calculated using a magnetisation factor $m=0.75$ and a Curie temperature $T_c=773$ °C, to calculate $B_s(T)$. The proeutectoid ferrite fraction increases almost linearly with decreasing temperature, with an increasing rate below about 730 °C. In figure 4.5 growth of the proeutectoid ferrite fraction as a function of temperature is given. The maximum proeutectoid ferrite fraction is 0.14.

In figure 4.6 the average particle radius as a function of temperature is given. In the beginning of the transformation (at a few percent ferrite) the particle radius is about 2 μm. It increases as the proeutectoid fraction increases. A peak in the particle radius can be seen at a ferrite fraction of about 0.16. In earlier experiments on steel with 0.60%C [1] a peak in the particle radius at a ferrite fraction of about 0.07 was observed. This indicates that this peak is related to the start in the pearlite transformation. Just before the δ peak the average particle radius is about 10 μm. After the formation of about 85% pearlite, there is a constant particle radius of 5 μm. The peak in δ is at 693 °C, whereas the start of the pearlite transformation seems to be at a slightly lower temperature, as can be seen in figure...
4.5. At temperatures above 740 °C the particle size is too small to be able to correctly calculate it. Therefore the particle sizes above this temperature in figure 4.6 are not really larger than at lower temperatures.

![Figure 4.7 Proeutectoid ferrite fraction versus average particle volume.](image)

Figure 4.7 Proeutectoid ferrite fraction versus average particle volume.

In figure 4.7 the proeutectoid ferrite fraction as a function of the average particle volume is given. Initially, the fraction increases strongly while the particle size remains very small. As soon as in figure 4.7 the increase in the particle volume is visible a linear increase in ferrite fraction versus particle volume can be seen. At a ferrite fraction of about 0.07 there is an increase in the rate of the ferrite fraction versus volume. After a proeutectoid ferrite fraction of 0.14 has been reached, the pearlite transformation starts.

![Figure 4.8 The quantity $\gamma_Y$ versus temperature.](image)

![Figure 4.9 Average of the square of the $y$-component of the magnetic induction versus temperature.](image)

Figure 4.8 The quantity $\gamma_Y$ versus temperature.

Figure 4.9 Average of the square of the $y$-component of the magnetic induction versus temperature.

In figure 4.8 and 4.9 the quantity $\gamma_Y$ and the average of the square of the $y$-component of the magnetic induction, derived from equations (2.36 and 2.32) are given as a function of temperature. The increase of the proeutectoid ferrite fraction increases $\gamma_Y$ and $<n_Y^2>$. 

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The pearlite transformation also has a large effect on $<n^2_y>$. At the end of the transformation $\gamma$ is about 0.25 and $<n^2_y>$ is about 0.85.

Figure 4.10  Microstructure of the transformed C45 sample, 200x.

Figure 4.11  Cross-section of the microstructure of the transformed sample C45, 200x.
In figure 4.10 and 4.11 the microstructure of the sample is given. The proeutectoid ferrite grain diameter is about 12 \( \mu \text{m} \) and the pearlite colony diameters are about 21 \( \mu \text{m} \). The pearlite has fine lamellar distances, of about 300 nm. Although the sample has been cooled very slowly there is still fine pearlite because of the alloying elements [2]. However at some points the pearlite has started to globularise.
4.2 Isochronal transformation experiments on 0.35% C steel (C35)

In section 4.2.1 the isochronal experiment on sample C35 is described. To be able to discuss the magnetic behaviour of the sample at different stages in the transformation, sweepfield experiments (see section 3.4) were carried out. These results are given in section 4.2.2. The effect of the wavelength of the neutrons at which the experiment has been carried out is shown in section 4.2.3, for an experiment which was done on the mirror polarimeter (SP) using neutrons with a shorter wavelength.

4.2.1 Transformation

Sample 35-1 was held at an austenisation temperature of 875 °C for one and a half hour and then cooled according to the temperature course given in figure 4.1. The applied external field was constant field of 680 A/m. The experiment was carried out on the crystal polarimeter with a sample environment as is given in figure 3.10. The thickness of the sample is 0.28 mm.

In figure 4.12 the rotation as a function of temperature is given. From the start of the transformation, at about 770 °C until about 710 °C there is a linear increase in the rotation. The rotation is still increasing at the end of the experiment at about 600 °C.

In figure 4.13 the depolarisation versus temperature is given. The depolarisation is dependent of $B_t^2$ (see equations (2.25) and (2.32) and therefore the depolarisation is more sensitive to temperature than the rotation. $T_g$ in the experiment can be best defined from the depolarisation, in this case this will be 770 °C. As $T_g$ in the heating experiment and $T_g$ in the cooling experiment seem to be at the same temperature, this will be the Curie temperature. For further calculations a Curie temperature of 770 °C is used.

![Figure 4.12 Rotation as a function of temperature.](image1)

![Figure 4.13 Logarithm of the determinant of the depolarisation matrix as a function of temperature.](image2)
The transformed samples were examined by quantitative image analysis to define the proeutectoid ferrite fraction. From this proeutectoid ferrite fraction the total ferrite fraction was calculated, in the same manner as for sample 45-2 (see section 4.1). In table 4.2 the values for the isochronal experiment on C35 is given.

Table 4.2 Fractions derived from CBA measurements on the transformed C35 samples.

<table>
<thead>
<tr>
<th></th>
<th>fraction proeutectoid ferrite</th>
<th>total fraction ferrite</th>
</tr>
</thead>
<tbody>
<tr>
<td>35-1</td>
<td>0.46</td>
<td>0.93</td>
</tr>
</tbody>
</table>

In figure 4.14 the ferrite fraction, as calculated from the cooling experiments, as a function of temperature is given. The fraction was calculated using a magnetisation factor $m=0.51$ and a Curie temperature $T_c=770$ °C. Now it is more clear that $T_p$ was indeed the Curie temperature, as there is a slight jump in the fraction at the starting temperature. Most probably above the Curie temperature the proeutectoid ferrite fraction increases very slowly in the beginning. At a certain point the transformation rate will increase towards the linear increase in the proeutectoid ferrite fraction which can be seen in figure 4.15. The maximum proeutectoid ferrite fraction is about 0.22.

![Figure 4.14](image1.png)  ![Figure 4.15](image2.png)

Figure 4.14 Ferrite fraction versus temperature.  Figure 4.15 Enlarged part of figure 4.14, proeutectoid ferrite fraction versus temperature.

In figure 4.15 the average particle radius as a function of temperature is given. The proeutectoid ferrite particle radius increases until about 13 µm at 720 °C. It seems as if there is no further growth of the particles just before the start of the pearlite transformation at the δ-peak at 693 °C. In the region between 693 and 720 °C there is however the same increase in proeutectoid ferrite fraction as there is in the temperature region of 770 to 720 °C. At the end of the pearlite transformation the average particle radius is about 4.7 µm.
In figure 4.17 the proeutectoid ferrite fraction as a function of average particle volume is given. There is a jump at the beginning of the transformation. It seems as if there is 8% of ferrite before the particle starts to grow. From figure 4.15 it is visible in the beginning of the transformation there is a rapid increase in fraction, because the formation of proeutectoid ferrite is started at a temperature above the Curie temperature. This is about 5% of ferrite. So before the particle volume is large enough to be able to see it in figure 4.17, there is about 5% ferrite which has been formed above $T_c$ and about 3% ferrite which is formed below $T_c$. The rate of increase in particle volume decreases as the ferrite fraction increases, as it is in other cases.

In figure 4.18 $\gamma_D$ is given as a function of temperature. An increase in the proeutectoid ferrite fraction has almost no effect on $\gamma_D$. At the start of the pearlite transformation there is an increase in $\gamma_D$, but as the pearlite fraction increases it slowly decreases again. In figure 4.19 the average of the square of the $y$-component of the magnetisation, $\langle n_y^2 \rangle$, is given. In decreasing the temperature the proeutectoid ferrite fraction increases, and $\langle n_y^2 \rangle$
increases. Further the course is the same as $\gamma$, as a function of temperature, a jump at the start of the pearlite transformation and a decrease towards lower temperatures.

Figure 4.18 The quantity $\gamma$ versus temperature.

Figure 4.19 Average of the square of the local magnetic induction direction versus temperature.

Figure 4.20 Microstructure of sample 35-1 after transformation, 200x.

In the figures 4.20 and 4.21 the microstructure of the transformed sample C35 (KP, constant field) is given. The average ferrite grain diameter is 25.7 $\mu$m and the average pearlite colony diameter is 27.2 $\mu$m. One can clearly see the rolling texture in figure 4.20, the distance between the bands is 29 $\mu$m. The dark pearlite has cementite with high
concentrations of manganese. The manganese stabilises the pearlite, so that it will not globularise easily.

Figure 4.21 Microstructure of a cross-section of sample 35-1 after transformation, 200x.

4.2.2 Magnetic behaviour

An isochronal sweepfield experiment has been carried out, similar to the one described in 4.2.1. In this experiment a sawtooth-like field, between -678.6 and +678.6 A/m, over 50 channels has been measured. Every second a saw-tooth was imposed. The sample was held at an austenisation temperature of 875 °C for one and a half hour and then cooled according to the temperature course given in figure 4.1. This experiment, on sample 35-2, has been carried out on the crystal polarimeter, with a sample environment as given in figure 3.10. The thickness of the sample was 0.285 mm.

The transformed sample was examined by quantitative image analysis to define the proeutectoid ferrite fraction. In table 4.3 the measured proeutectoid fraction and the calculated total ferrite fraction are given.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Fraction proeutectoid ferrite</th>
<th>Total fraction ferrite</th>
</tr>
</thead>
<tbody>
<tr>
<td>35-2</td>
<td>0.42</td>
<td>0.93</td>
</tr>
</tbody>
</table>

Table 4.3 Fractions derived from Quantimet measurements on the transformed C35 sample.
In figure 4.22 the rotation as a function of temperature is given for the maximum magnetic field, half of the maximum magnetic field and for almost zero field.

![Figure 4.22 Rotation as a function of temperature.](image1)

In figure 4.23 the logarithm of the determinant of the depolarisation matrix is given as a function of temperature. During the formation of proeutectoid ferrite the depolarisation increases very rapidly.

![Figure 4.23 Logarithm of the determinant of the depolarisation matrix as a function of temperature, at maximum field.](image2)

In figure 4.24 the ferrite fraction, as derived from the cooling experiments, as a function of temperature is given. For the maximum field, a magnetisation factor $m=0.51$ and a Curie temperature $T_c=770{\,}^\circ{\text{C}}$ have been used. These are the same values as used in the

![Figure 4.24 Ferrite fraction as a function of temperature, for maximum applied field.](image3)

![Figure 4.25 Average particle size as a function of temperature, for three different field values.](image4)
calculation of the ferrite fraction for the sample described in 4.1.1. In figure 4.25 the average particle radius, as derived from the experiments, as a function of temperature is given, for three different values of the applied magnetic field. The maximum proeutectoid particle size ranges from 9 \( \mu \text{m} \) measured in zero field, to 12 \( \mu \text{m} \) measured at 678.6 A/m. At 697 °C the pearlite transformation starts, as indicated by the \( \delta \) peak. After the pearlite transformation the average particle size is about 4 \( \mu \text{m} \).

In figures 4.26 until 4.29 the hysteresis curves at different stages of the transformation are given for the hysteresis in rotation, average particle size and \( \gamma \). Figure 4.26 gives the situation at the start of the transformation. In figure 4.26a the rotation is given as a function of applied field, there is almost no coercivity at the start of the transformation. The ferromagnetic material in the sample seems to be saturated, as the rotation at larger field (500-700 A/m) seems to be constant. While the proeutectoid ferrite fraction increases, also the coercivity increases. In figure 4.26b the average particle size is given as a function of applied field, there is no clear dependence of the particle size on the applied field. In figure 4.26c the quantity \( \gamma \) is given. Here also no clear dependence is visible.

In figure 4.27 the situation at the \( \delta \) peak is given. The microstructure at the start of the pearlite transformation has a higher coercivity than in the beginning of the proeutectoid ferrite transformation. In figure 4.27b and c there is now hysteresis in the particle size and in \( \gamma \). Both increase linearly with applied field.

In figure 4.28 the hysteresis curves for a sample of about 25% proeutectoid ferrite, 50% pearlite and 25% austenite is given. There is no longer saturation of the ferromagnetic material at the higher fields. The coercivity is hard to determine, but will be higher than in figure 4.28a. The average particle size and \( \gamma \) are both difficult to determine, as it is difficult to determine the exact curves in the pearlite transformation.

In figure 4.29 the hysteresis curves at the end of the transformation is given. From figure 4.29a one can see that there is no saturation, and the coercivity is about 95 A/m. At high fields (500-570 A/m) the average particle size is almost constant, around zero field there is a difference the particle size is large with decreasing field, and almost constant with increasing field. The same behaviour can be seen in \( \gamma \).
Figure 4.26a Rotation as a function of applied external field at the beginning of the proeutectoid ferrite transformation.

Figure 4.27a Rotation as a function of applied external field at the beginning of the pearlite transformation.

Figure 4.26b Average particle radius as a function of applied external field.

Figure 4.27b Average particle radius as a function of applied external field.

Figure 4.26c The quantity $\gamma$, as a function of applied external field.

Figure 4.27c The quantity $\gamma$, as a function of applied external field.
Figure 4.28a Rotation as a function of applied external field, in the pearlite transformation.

Figure 4.28b Average particle radius as a function of applied external field.

Figure 4.28c The quantity $\gamma_y$ as a function of applied external field.

Figure 4.29a Rotation as a function of applied external field, at the end of the pearlite transformation.

Figure 4.29b Average particle radius as a function of applied external field.

Figure 4.29c The quantity $\gamma_y$ as a function of applied external field.
In figure 4.30 the coercivity is plotted as a function of temperature. At the end of the proeutectoid ferrite transformation the coercivity is about 36 A/m, whereas in the beginning this is 20 A/m. There is a small peak in the coercivity at the temperature where there is a peak in particle size, but the hysteresis curves in the pearlite transformation are very difficult to determine. Maybe there is an increase in the coercivity, starting from 697 °C.

Figure 4.30 Coercivity as a function of temperature

In figure 4.31 the remanent rotation is given as a function of temperature. The remanence is zero until about 750 °C, while with increasing proeutectoid ferrite fraction the remanence increases slowly until the start of the pearlite transformation. During the pearlite transformation $\phi_r$ increases more rapidly.

Figure 4.31 Remanence as a function of temperature.

It is possible to fit a line of $\phi = b_0 + b_1 H$ through the hysteresis curves at high fields (±500-750 A/m). $b_1$ and $b_0$ give direct information about the saturation induction of the sample. If there is saturation, $b_1$ will be zero. In figure 4.32 the average value of the fit at positive and negative field value of $b_0$ is given. Increasing the proeutectoid ferrite fraction
increases the rotation, what can be seen in the gradual increase in \( b_0 \). Increasing the pearlite fraction gives a further increase in \( b_0 \). In figure 4.33 the average value of the fit at positive and negative field of \( \frac{b_1}{\phi} \) is given. If \( \frac{b_1}{\phi} \) is zero there is fully saturation, the value of \( \frac{b_1}{\phi} \) above 750 °C is about \( 5 \cdot 10^{-6} \) rad m/A, only from 750 °C there is an increase. This means that until the start of the pearlite transformation the ferromagnetic material is almost saturated. In the situation at the end of the transformation \( \frac{b_1}{\phi} \) is about 0.007 rad m/A.

![Figure 4.32 Value of the fit of \( b_0 \).](image1)

![Figure 4.33 Value of the fit of \( \frac{b_1}{\phi} \).](image2)

In the figures 4.34 and 4.35 \( \gamma_r \) and the average of the square of the y-component of the magnetisation as a function of temperature are given for maximum applied field. Both increase with increasing proeutectoid ferrite fraction, but \( < n_y^2 > \) increases faster than \( \gamma_r \). After the pearlite transformation \( \gamma_r \) is about 0.5 and \( < n_y^2 > \) is about 0.85.

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Figure 4.34 The quantity $\gamma$, versus temperature, for maximum applied field versus temperature.

Figure 4.35 Average of the square of the y-component of the magnetisation for maximum applied field versus temperature.

In figures 4.36 and 4.37 the microstructure of the transformed sample 35-2 is given.

Figure 4.36 Microstructure of sample 35-2 after transformation, 200x.
Figure 4.37  Microstructure of a cross-section of sample 35-2 after transformation, 200x.
4.2.3 Wavelength dependency

In the mirror polarimeter experiments have been carried out using a wavelength of 0.123 nm. Sample 35-5 has been austenitised at 860 °C for one and a half hour. Then the same experiment as described in section 4.2.1 was performed. The only difference is that the sample configuration with rectangular samples (see figures 3.11 and 3.12) was used instead of the ring-shaped samples. This experiment has been carried out at a field of 5440 A/m. The sample thickness was 0.42 mm.

The results of this experiment were difficult to fit. This is the result of the use of the sample holder with iron rings to provide flux closure. In section 3.3 this is discussed.

In figure 4.38 the rotation as a function of temperature is given. Because of the larger fields in this sample configuration, the total rotation is much larger than in the KP experiment. In figure 4.39 the logarithm of the determinant of the depolarisation matrix is given as a function of temperature.

![Figure 4.38 Rotation as a function of temperature.](image)

![Figure 4.39 Logarithm of the determinant of depolarisation matrix as a function of temperature.](image)

The transformed sample has been analysed by quantitative image analysis, in table 4.4 the results are given. To get a total ferrite fraction of 0.93 a magnetisation factor $m=0.91$ and a Curie temperature $T_c=770$ °C have been used.

| Table 4.4 Fractions derived from Quantimet measurements on the transformed C35 sample. |
|---------------------------------|-----------------|-----------------|
| fraction proeutectoid ferrite | total fraction ferrite |
| 35-5                           | 0.45             | 0.93            |

In figure 4.40 the ferrite fraction, as calculated from the cooling experiment, as a function of temperature is given, for both the KP ($\lambda=0.16$ nm) and SP ($\lambda=0.123$ nm) experiment. The difference in temperature and the irregularities in the curve for the SP experiment are
all explained in section 3.3. The global trend of the curve can however be compared to the curve of the KP experiment. The most clear difference can be seen in the proeutectoid ferrite fraction, as derived from the results. In the KP experiment the value as derived from the results is 0.22 and the value obtained from image analysis is 0.46. In the SP experiment the proeutectoid ferrite fraction, as calculated from the experiment is about 0.43 and the value obtained from image analysis is 0.45.

In figure 4.41 the average particle radius as a function of temperature for both the KP and SP experiments are given. The maximum proeutectoid ferrite particle size, as calculated from the experiment is larger for the SP experiment than for the KP experiment. In the KP experiment the particle radius increases during cooling to about 730 °C and then there is a kind of plateau level in the particle radius which persists until the start of the pearlite transformation. Also in the SP experiment a plateau is visible, but at 25 μm instead of at 13 μm in the KP experiment. After the δ peak in the KP experiment there is a gradual decrease in particle radius, whereas in the SP experiment there is an increase in particle radius. At 600 °C the particle radius in the KP experiment is 4.5 μm, and the particle radius in the SP experiment is about 17 μm.

In figures 4.42 and 4.43 the microstructure of the transformed sample 35-5 is given.
Figure 4.42  Microstructure of sample 35-5 after transformation, 200x.

Figure 4.43  Microstructure of a cross-section of sample 35-5 after transformation, 200x.
4.3 Isochronal transformation experiments on 0.22% C steel

During this experiment sample 22-1 was held at an austenisation temperature of 880 °C for one and a half hour and then cooled according to the temperature course given in figure 4.1. The applied external field was a constant field of 680 A/m. The experiment has been carried out in the crystal polarimeter, the sample configuration was that of figure 3.10.

In figure 4.44 the rotation is plotted versus temperature. There is a jump in the rotation at 773 °C, which indicates that this is the Curie temperature. The transformation of austenite to ferrite has begun before the ferrite was ferromagnetic, as is expected. The rotation seems to increase to a certain plateau level until the start of the pearlite transformation. In figure 4.45 the logarithm of the determinant of the depolarisation matrix as a function of temperature is given. From $T_\varphi$ to 750 °C there is almost no difference between the cooling and the heating curve.

The transformed samples were examined by quantitative image analysis, in this way the proeutectoid ferrite fraction was determined. In table 4.5 these fractions are given.

<table>
<thead>
<tr>
<th>Table 4.5 Fractions derived from CBA measurement on the transformed C22.</th>
</tr>
</thead>
<tbody>
<tr>
<td>22-1</td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

In figure 4.46 the ferrite fraction, as calculated from the cooling experiments, as a function of temperature is given. The fraction was calculated using a magnetisation factor of 0.43 and a Curie temperature of 773 °C, to get a total ferrite fraction of 0.98. Compared to the rotation there is a more pronounced jump in the fraction at the Curie temperature. If the right correction is made with $B_s(T)$, this jump would be a vertical line. From $T_\varphi$ to about...
765 °C, the calculated ferrite fraction is not showing the real fraction at that temperature. The proeutectoid ferrite fraction formed above \( T_c \) is 0.33. Just below 700 °C the pearlite transformation starts and in only a few degrees the total pearlite transformation is finished. From figure 4.46 it seems as if there is only growth of proeutectoid ferrite at temperatures above 730 °C. The maximum proeutectoid ferrite fraction is 0.60.

Figure 4.46 Ferrite fraction versus temperature.

In figure 4.47 the particle radius, as derived from the results, as a function of temperature is given. Only below 765 °C this gives a view of the austenite to ferrite transformation. At 760 °C a maximum particle radius of 16 \( \mu m \) is reached. At this point the pearlite transformation has not yet started, and the proeutectoid ferrite fraction is still increasing. After the maximum, the particle radius decreases to about 10 \( \mu m \), while the ferrite fraction increases.

Figure 4.47 Average particle radius versus temperature.

In figure 4.48 the proeutectoid ferrite fraction as a function of the particle volume is given. From a ferrite fraction of 0.3 to 0.4 there is an increase in the particle volume with increasing fraction. From a fraction of 0.4 until the start of the pearlite transformation an
decreasing rate in the particle volume with increasing fraction can be seen. This would be expected on base of the decreasing particle size below 760 °C (figure 4.47).

In figures 4.49 and 4.50, $\gamma_y$ and the average of the square of the y-component of the magnetisation as a function of temperature are given. It seems as if there is a maximum in both $\gamma_y$ and $<n_y^2>$ at 745 °C. at this temperature the ferrite fraction starts to form with a decreasing rate. Both parameters increase at the start of the pearlite transformation. At the end of the transformation $\gamma_y$ is decreased to about 0.42 and $<n_y^2>$ to 0.74.

![Figure 4.49](image1.png)  
![Figure 4.50](image2.png)

Figure 4.49 The quantity $\gamma_y$ versus temperature.  
Figure 4.50 Average of the square of y-component of the magnetisation versus temperature.

In figure 4.51 and 4.52 the microstructure of the transformed sample is given. The average ferrite grain diameter is about 30 μm and the average pearlite colony diameter is about 20 μm.
Figure 4.51  Microstructure of sample C22 after transformation, 200x.

Figure 4.52  Microstructure of a cross-section of the C22 sample, 200x.
5 Discussion

5.1 Transformation

In figure 5.1 the fraction ferrite as a function of temperature is given for the cooling curves of samples 45-2, 35-2 and 22-1. The rate of formation of proeutectoid ferrite is linear with temperature in the case of C35, and non-linear in the case of C45. For C22 only between 765 °C and 730 °C one can see the proeutectoid ferrite growth. At higher temperatures the ferrite is not yet ferromagnetic, and at lower temperatures it seems as if there is a limit value to the proeutectoid ferrite fraction one can measure. There is a maximum in the ferrite fraction for sample C22 at about 660 °C, this is caused by the choice of \( B(T) \), and indicates that a different curve for \( B(T) \) would be better.

![Ferrite fraction versus temperature for the three different steel compositions.](image)

\( T_\varphi \) can be the \( A_3 \) temperature or the Curie temperature. From the jump in the fraction transformed at \( T_\varphi \) of C22 and C35 one can conclude that this has to be the Curie temperature. These temperatures are compared to the Curie temperatures calculated by MTDATA in table 5.1. For C22 also the Curie temperature as measured by DTA is given. The Curie temperatures as measured by neutron depolarisation are much higher than the calculated Curie temperatures. For the calculation with MTDATA the composition of the ferrite was used, assuming the manganese to be homogeneously distributed over the different phases. From the final microstructures it can be seen that the manganese was partially located in the cementite of the pearlite. Therefore, the manganese content in the ferrite phase is lower than the overall content, the manganese concentration in the ferrite, that was used for the MTDATA calculation, was too large giving a \( T_c \) that is too low.

Increasing the manganese concentration decreases the Curie temperature by 15K per percent Mn. The measurements of the DTA seem not to be reliable because of the hysteresis in Curie temperature in the heating and cooling curves, see section 3.1.3.

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Table 5.1 $T_\phi$, the measured $T_c$ temperature and the calculated $T_c$ temperature.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>$T_\phi$ in °C</th>
<th>$T_c$ as measured by DTA in °C</th>
<th>$T_c$ as calculated by MTDATA in °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>22-1</td>
<td>773</td>
<td>756</td>
<td>761</td>
</tr>
<tr>
<td>35-1</td>
<td>770</td>
<td>-</td>
<td>758</td>
</tr>
<tr>
<td>45-2</td>
<td>761 (A₃)</td>
<td>-</td>
<td>759</td>
</tr>
</tbody>
</table>

If one compares the total ferrite fractions, as measured by quantitative image analysis and calculated by MTDATA, they do not differ much, as can be seen in table 5.2. Only C22 may be slightly decarburated, because the total ferrite fraction is higher than is expected. If one checks the microstructure at different cross-sections of all samples one sees that there is a difference between the structure at the surface and the structure in the centre of the sample. This indicates a ferrite layer, which could be caused by surface-enhanced nucleation. This ferrite layer will not contribute to the depolarisation, because it is expected to magnetise homogeneously, and depolarisation only arises from differences in magnetisation over the sample area.

Table 5.2 Total ferrite fraction, measured and calculated.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Total ferrite fraction, measured by quantitative image analysis</th>
<th>Total ferrite fraction, calculated by MTDATA</th>
</tr>
</thead>
<tbody>
<tr>
<td>22-1</td>
<td>0.98</td>
<td>0.96</td>
</tr>
<tr>
<td>35-1</td>
<td>0.93</td>
<td>0.93</td>
</tr>
<tr>
<td>35-2</td>
<td>0.93</td>
<td>0.93</td>
</tr>
<tr>
<td>35-5</td>
<td>0.93</td>
<td>0.93</td>
</tr>
<tr>
<td>45-2</td>
<td>0.90</td>
<td>0.91</td>
</tr>
</tbody>
</table>

Table 5.3 Measured values of the proeutectoid ferrite fraction compared to the values as derived from the experiments.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>proeutectoid ferrite fraction, measured by quantitative image analysis</th>
<th>proeutectoid ferrite fraction, calculated from the ND experiments</th>
<th>proeutectoid ferrite fraction, calculated by MTDATA</th>
</tr>
</thead>
<tbody>
<tr>
<td>22-1</td>
<td>0.84</td>
<td>0.60</td>
<td>0.71</td>
</tr>
<tr>
<td>35-1</td>
<td>0.46</td>
<td>0.22</td>
<td>0.47</td>
</tr>
<tr>
<td>35-2</td>
<td>0.42</td>
<td>0.25</td>
<td>0.47</td>
</tr>
<tr>
<td>35-5</td>
<td>0.45</td>
<td>0.46</td>
<td>0.47</td>
</tr>
<tr>
<td>45-2</td>
<td>0.16</td>
<td>0.14</td>
<td>0.31</td>
</tr>
</tbody>
</table>

The maximum proeutectoid ferrite fraction has been measured by quantitative image analysis on the final microstructures. This has also been calculated by MTDATA. To calculate the ferrite fraction from the rotation in the neutron depolarisation experiments only the total ferrite fraction is used to determine $m$. The proeutectoid ferrite fraction, as calculated from the experiments, can then be compared to the measured proeutectoid fraction. In table 5.3 these results are given. Only for sample 35-5 and 45-2 the
proeutectoid ferrite fraction as calculated from the neutron depolarisation experiments equals the proeutectoid ferrite fraction as measured by quantitative image analysis.

Reducing the initial carbon concentration leads to more depolarisation in the proeutectoid ferrite phase. In the curves of figure 5.1 and 5.2 this can be best seen in the worse statistics of sample C22 compared to C35 and C45.

![Figure 5.2](image)

Figure 5.2  Average particle radius versus temperature for the three different steel compositions.

In figure 5.2 the average particle radii for the three isochronal experiments are given. The decrease in \( \delta \) at temperatures between 760°C and 700 °C for C22 is physically only possible if the particles divide into multidomains, there is nucleation of new particles, or if the total rotation per domain is too large to be measured. In table 5.4 the ferrite and pearlite particle radii determined from the microstructure and experiment are compared. The pearlite particle radius is measured from the microstructure by taking the radius of the pearlite colonies. The “pearlite particle radius” is the average particle radius of ferrite and pearlite. For all samples the ratio of proeutectoid ferrite to pearlite is different, therefore this cannot be compared to one another, and not to the pearlite particle radius as measured from the microstructure.

Table 5.4 Particle radii as determined from the microstructure and the experiments.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>ferrite particle radius determined from the microstructure [µm]</th>
<th>ferrite particle radius ND [µm]</th>
<th>pearlite particle radius determined from the microstructure [µm]</th>
<th>pearlite particle radius ND [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>22-1</td>
<td>15.0</td>
<td>16.0</td>
<td>10.0</td>
<td>4.0</td>
</tr>
<tr>
<td>35-1</td>
<td>12.9</td>
<td>13.0</td>
<td>13.6</td>
<td>4.7</td>
</tr>
<tr>
<td>45-2</td>
<td>6.0</td>
<td>10.0</td>
<td>10.5</td>
<td>5.0</td>
</tr>
</tbody>
</table>

The ferrite particle radius seems to be rather well measured in the ND experiment for samples 22-1 and 35-1, except for sample 45-2. But from figure 5.2 one would conclude
that the particle radius of C22 is not correct. Probably the calculation of the particle radius is not correct. In the case of C22 one would expect the particle radius to increase with decreasing temperature, until the start of the pearlite transformation, so the maximum proeutectoid ferrite particle radius would be larger than 16 µm. This would mean that the particle radius, as measured from the experiments, is larger than in reality.

The particle size, as calculated from the experiments would give an incorrect value if (some) particles were divided into several domains. If every particle is divided into 2 or 4 domains like in figure 5.3, the mean measured particle diameter is 0.62 of the true particle diameter.

![Figure 5.3 Multidomain particles.](image)

The δ peak gives an indication of the start of the pearlite transformation, therefore the peak will be compared to the A₁ temperature. There is a difference in the pearlite start temperature between C22 and the other two samples, as is visible in figure 5.2. In table 5.5 the temperature at which the δ peak is situated is compared to the A₁ temperature, as calculated by MTDATA. The temperatures at which the δ peak is situated is lower than the calculated A₁ temperature, because in the experiment there is no local equilibrium. A certain undercooling is necessary.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>δ peak temperature in °C</th>
<th>A₁ temperature calculated by MTDATA in °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>22-1</td>
<td>700</td>
<td>717</td>
</tr>
<tr>
<td>35-1</td>
<td>692</td>
<td>721</td>
</tr>
<tr>
<td>45-2</td>
<td>693</td>
<td>722</td>
</tr>
</tbody>
</table>

In figure 5.4 the ferrite fraction as a function of average particle volume is given, for samples 35-1 and 45-2. C45 has been measured from the beginning of the transformation, the transformation of C35 started above the Curie temperature. In the beginning of the transformation the particle volumes are too small to be measured, but these small particles can give a ferrite fraction of up to 0.08 in the case of C35. C35 first shows an increase in
volume compared to a small increase in ferrite fraction, and from a volume of $0.9 \times 10^{-4}$ \(\mu m^3\) a larger increase in ferrite fraction as compared to the particle volume. The curve for sample C45 has a larger first increase in ferrite fraction as compared to particle volume than sample C35, and the curve changes to an even larger increase in ferrite fraction as compared to the particle volume at $0.2 \times 10^{-4}$ \(\mu m^3\).

![Figure 5.4 Proeutectoid ferrite fraction as a function of average particle volume for C35 and C45.](image)

In the figures 5.5 and 5.6 the two experiments on the crystal polarimeter on C35 are compared. The rotation as a function of temperature is given for the entire transformation in figure 5.4, and for the proeutectoid transformation in figure 5.5. Sample 35-1 was measured at a constant field, and sample 35-2 at a varying field. The start of the experiment is exactly the same in temperature for both samples. The rotation as a function of temperature in the proeutectoid region seems to be equal. There is a difference in the pearlite transformation, however. This is a difference in transformation temperature and a difference in the rotation at the end of the pearlite transformation. The reproducibility of the experiments is therefore not perfect, but at least in the proeutectoid region fairly good.

![Figure 5.5 Rotation as a function of temperature for the experiments on samples 35-1 (constant field) and 35-2 (varying field).](image)

![Figure 5.6 Enlargement from figure 5.5.](image)
5.2 Magnetic behaviour

The cooling experiments start from a magnetically saturated situation, whereas the heating experiments start from a non-magnetised situation, where there is already ferromagnetic material present. That is why the maximum rotation in the heating experiments is lower than in the cooling experiments. Only in experiment C22 this seems not to be the case. There is an uncertainty in the rotation angle at temperatures below the pearlite transformation. The rotation here could be $2\pi$ radians higher or lower than as presented in figure 4.43. A sweepfield experiment would give a better insight into this.

The crystal anisotropy increases with decreasing temperature. In the heating experiment of sample C45 this can be seen. Increasing the temperature would decrease the rotation, as the ferrite will transform to austenite. In figure 4.2 an increase of the rotation can be seen at the start of the heating curve.

In table 5.6 the magnetisation factors are given which are used to calculate the ferrite fraction. If $m=1$ the samples are saturated. Saturation has best been approached in sample 35-5, because this experiment has been carried out at 8 times higher fields than the other experiments. Besides that, the sample had a rolling texture in the direction of the applied field and therefore it was easy to magnetise. Samples 45-2 and 35-1 are less well magnetised. Sample 22-1 has a magnetisation factor less than 0.5, which would indicate that it is very badly magnetised. As the results of the C22 experiment raise a lot of questions, it will be very useful to repeat this experiment with a magnetic sweepfield. This will give a more reliable value of the magnetisation factor.

Table 5.6 Magnetisation factors used for the calculation of the ferrite fraction

<table>
<thead>
<tr>
<th>Sample code</th>
<th>m</th>
</tr>
</thead>
<tbody>
<tr>
<td>22-1</td>
<td>0.43</td>
</tr>
<tr>
<td>35-1</td>
<td>0.51</td>
</tr>
<tr>
<td>35-2</td>
<td>0.51</td>
</tr>
<tr>
<td>35-5</td>
<td>0.91</td>
</tr>
<tr>
<td>45-2</td>
<td>0.75</td>
</tr>
</tbody>
</table>

In the experiments with the samples 22-1 and 35-1 the maximum proeutectoid ferrite fraction, calculated from the results, was lower than what was found in the quantitative image analysis, as can be seen in table 5.3. This could also be a consequence of the use of a constant magnetisation factor during the whole experiment. If, in the proeutectoid ferrite growth region, a magnetisation factor decreasing with temperature was used, a higher maximum proeutectoid ferrite fraction would be achieved. In the figures 5.7 and 5.8 it is explained why a decreasing $m$ is realistic. At the beginning of the proeutectoid ferrite transformation there will be some ferromagnetic spheres in a paramagnetic matrix, and the magnetisation will be in the direction of the applied field. A possible (extreme) situation at the end of the proeutectoid ferrite transformation is drawn in figure 5.7. There are ferrite layers on the former austenite grain boundaries. The magnetisation will more likely be
directed in the plane of the grain boundaries. This situation will have a lower $m$ than in the beginning.

![Diagram showing grain boundaries and phases](image)

**Figure 5.7** Situation at the beginning of the proeutectoid ferrite formation.

**Figure 5.8** Situation at the end of the proeutectoid ferrite formation.

In all experimental results the peak in the particle radius corresponds with the early stages of the pearlite formation. This peak can be found in both the heating and cooling curves, and is smaller at lower carbon concentration. Because the particle radius is a calculated value, it is better to look at the correlation length $\xi$, as this is directly derived from the experiment. Also in $\xi$ this peak can be found. There is not yet an acceptable explanation for this phenomenon. Possible causes are described here.

In the situation of figure 5.8 one can see that there are paramagnetic regions, surrounded by ferromagnetic material. This would give an inhomogeneous situation, with large demagnetisation fields. Large inhomogeneities in a homogeneously magnetised material will give large correlation lengths, and therefore a peak in $\delta$. The fraction at which these surrounded paramagnetic regions would form would be more or less the same in different carbon steels (at the same austenite grain sizes). The experiments however show that the $\delta$ peak depends on the carbon content, and therefore on the temperature at which the pearlite starts to form. If the austenite grains transform to pearlite one after one, then there are also large inhomogeneities present, which would give large correlation lengths.

If the pearlite is nucleated on an austenite grain boundary, this will give demagnetisation fields, as the fluxlines through the ferrite in pearlite are not able to pass through the paramagnetic austenite. Pearlite nucleated on proeutectoid ferrite would not give these demagnetisation fields. It might be possible that the $\delta$ peak arises because of these demagnetisation fields. Sample C22 has a larger fraction of proeutectoid ferrite fraction than sample C35 and C45. The chance of pearlite nucleating on proeutectoid ferrite instead of austenite will therefore be larger in C22 than in C35 and C45. This would give a
larger δ peak in C45 than in C35 and larger in C35 than in C22. In figure 5.2 this effect is visible.

From figure 4.30 one can see that the coercivity increases with increasing proeutectoid ferrite fraction. Because grain boundaries are formed, the density of the pinning sites increases, and therefore the coercivity increases. From the start of the pearlite transformation there is an increase in coercivity because a magnetic complex structure is formed with many pinning possibilities at the cementite lamellae, the pearlite colony boundaries, and the ferrite/pearlite boundaries.

The coercivity in the proeutectoid ferrite phase is rather low (20-30 A/m). This would indicate that there are multidomain particles. In single domain particles it is difficult to turn the direction of the magnetic moment into the direction of the applied field, which will lead to a high coercivity. In multidomain particles the domain with the direction of the magnetic moment closest to the applied field can grow at a cost of the other domain(s). If the particle has not yet become a single domain before the direction of the applied field is changed, then this is a relatively easy process and therefore the material has a lower coercivity.

![Diagram](image.png)

Figure 5.9 Schematic hysteresis curves for the proeutectoid ferrite phase and the pearlitic phase.

It is possible that the hysteresis curve at 50% ferrite fraction is a summation of two hysteresis curves, as given in figure 5.9. The slope of the hysteresis curve gives the demagnetisation factor \( N_d \). From the demagnetisation factor the dimensions of the ferrite and cementite plates can be found.
In figure 5.10 the quantity $\gamma_y$ is given for the three different steel samples. Increasing the proeutectoid ferrite fraction slightly increases $\gamma_y$. As soon as there are more particles, they will influence each other, and therefore they will align better in the field direction.

There is a difference in $\gamma_y$ in the pearlitic phase between the curves of C22 and C35 on the one hand and C45 on the other. $\gamma_y$ for C45 is smaller, which would indicate that the pearlite has more fluctuations in the magnetisation in sample C45. C35 and C22 have a microstructure with more or less a proeutectoid ferrite matrix with pearlite colonies in that matrix. Sample C45 has a homogeneously distributed ferrite/pearlite composition, and could therefore have a lower $\gamma_y$.

The $\gamma_y$ curve of sample C22 shows a local maximum at 745 °C, this may indicate the start of the multidomain particles. In decreasing the temperature the particles will grow, while the correlation lengths become smaller.

### 5.3 Wavelength effect

The formulas given in section 2.3 are derived by using the small-angle approach [20]. It is assumed that the Larmor precession angle in each domain is much smaller than 1. At larger angles the applied formulas are no longer valid. Neutron depolarisation is seen as the result of small-angle scattering of a polarised neutron beam by magnetic inhomogeneities. It is assumed that the inhomogeneities are so large that scattering occurs only at small angles and all neutrons enter the analyser. The angle of rotation per particle should be sufficiently small and in any case smaller than $\pi/2$ radians. Equation (2.13) for the rotation angle can be written as:

$$\varphi = B_L L_y \sqrt{2.18 \cdot 10^{29} \lambda},$$  \hspace{1cm} (5.1)

and so giving the wavelength dependency of the angle of rotation. From equation (5.1) it is clear that one can measure larger domains by using smaller wavelengths.
In figure 4.39 the fraction transformed measured on C35 with wavelength 0.16 and 0.123 nm are compared. The large difference in observed $T_e$ and in the rest of the temperature (10-20 °C) arises from the fact that the molybdenum coils were shielded in the KP experiment and not in the SP experiment, as explained in section 3.3. However a $T_e$ of 770 °C has been used to calculate $B_s(T)$, this is the same $T_e$ as was used for the other C35 samples. By taking a lower $T_e$, the calculation just below $T_e$ might be better. The maximum proeutectoid ferrite fraction with $\lambda=0.123$ nm is very close to the measured fraction by quantitative image analysis (see table 5.3), whereas the maximum proeutectoid ferrite fraction as measured with $\lambda=0.16$ nm is about half of the measured fraction by quantitative image analysis. If one compares the growth of the proeutectoid ferrite of sample C45 (figure 4.4) to the that of C35 given in figure 4.39, then it seems strange that there is a linear growth until the start of the pearlite transformation for the KP experiment on C35. Sample C45 and C35 in the SP experiment show an increasing rate of the proeutectoid ferrite growth close to the pearlite transformation.

In figure 4.40 the average particle radii for the SP and KP experiment on sample C35 are compared. In the SP experiment the particle radius seems to grow faster, and reaches a higher level before the pearlite transformation. As the sample is depolarised before the start of the pearlite transformation (see figure 4.38) the statistics are worse in this region, which can be seen in the cooling curve below 700 °C (figure 4.37). The peaks before the pearlite transformation in the particle size which is seen in figure 4.40 for the SP experiment are caused by these lower statistics.

The difference in $f$ and $\delta$ between the SP and the KP experiment can arise from the difference in wavelength, but also from the fact that the SP experiment was done at an applied field of 8 times higher. This will not give rotation of 8 times larger, as the magnetisation is already 0.75 and could not be higher than 1. Besides the effect of the applied field, there is also an effect because of the direction of the field compared to the direction of the rolling texture, as is explained in section 3.3. In the SP experiment the configuration of figure 3.11 and 3.12 was used, which is more easy to magnetise. As there were too many differences between the experiment on 35-1 and 35-5, it is not possible to make a distinction between the different effects.
6 Conclusions

Neutron depolarisation measurements can give important information about the fraction ferrite and particle size during the transformation. However, in translating the measured parameters to the ferrite fraction and particle size, there are still some difficulties. For example, it is not yet possible to obtain a reliable fraction and particle size after doing one experiment on a certain steel. Sweepfield experiments are necessary to be able to determine the maximum rotation after the transformation, whereas experiments with a constant field give more information about the transformation itself, because of a better time resolution and higher statistics. In doing experiments with varying field information on the magnetisation of the steel is obtained. This provides also possibilities to describe the microstructure at several stages of the transformation. Secondly, there is a strong influence of $B_s$ on $f$ and $\delta$. Maybe a $B_s$ calculated for medium carbon steels, for the temperature region of interest, in stead of a $B_s$ for pure iron would give better results.

Furthermore, in this work small cooling rates were used, therefore the particle sizes were relatively large. If the total rotation per particle becomes larger than $\pi/2$ radians this will not be measured. Both the ferrite fraction and the average particle size will then not be correctly determined. On the run-out cooling table in the industrial situation the cooling rates are much higher. This will give a microstructure with smaller particle sizes. If neutron depolarisation experiments are carried out at large cooling rates, this particle size problem will not be a problem. If, at some stage in the transformation, multidomain particles would arise, this will give a wrong calculation of $\delta$. It will be important to know if, and if so, when these multidomain particles are present.

In this work it is demonstrated that it is possible to perform, to a certain extent, reproducible experiments with neutron depolarisation. It is very important to have a reproducible sample environment. The orientation of the sample and the molybdenum coils with regard to the neutron beam must be the same for each experiment. Also the position of the thermocouple on the sample is important to really measure the sample temperature. This makes the design of the sample environment an important parameter for the quality of the experiment.

By doing experiments on different carbon steels, it is found that the peak in particle size is caused by the early stages of the pearlite transformation. Several possible causes of the $\delta$ peak have been presented.

The results of the experiments given in this work for C45 agree with the image analysis results. For sample C35 and C22 either the rotation per domain may be too large or there are multidomain particles, therefore the proeutectoid fraction measured with neutron depolarisation and with image analysis does not agree. These samples could however be measured accurately if the experiment would be carried out at larger cooling rates, with smaller final particle sizes.
Acknowledgements

During my graduation period I have been encouraged and helped by a lot of people, at both the Interfaculty Reactor Institute and the Laboratory of Materials Science. First of all I would like to thank Suzanne te Velthuis for teaching me the technique of neutron depolarisation, helping to do experiments, processing data and commenting this report. My first steps into magnetisation were guided by Theo Rekveldt. He has helped a lot in the interpretation of the results, combined with all possible magnetisation phenomena. My third supervisor, Jilt Sietsma, I would like to thank for discussing all the results and showing me all the interesting problems I encountered. Professor van der Zwaag has provided “back-up” support during this graduation period, but more important, he has made me enthusiastic for the steel research.

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References


