In Situ Detection and Characterisation of Phase Transformations in Weld Metals

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The work presented in this thesis is concerned with the in situ detection of phase transformations in weld metals. In order to obtain favourable mechanical properties of a material it is useful to map its phase transformation behaviour. Cooling rate and chemical composition are factors of great influence to the transformation behaviour. For a given composition, a continuous cooling transformation (CCT) diagram can be used to illustrate this behaviour for a number of cooling rates. Usually these diagrams are constructed from data obtained through dilatometry, which is an expensive and, in the case of welding, not always accurate method.

The aim of this work was to develop a new methodology and a set of tools for the construction of weld metal CCT-diagrams. The methodology is based on analysis of weld thermal histories acquired from temperature measurements in the fusion zone.

The temperature was measured with thermocouples, logged with LabView and analysed numerically. The microstructure was characterised with light optical microscopy and field emission gun scanning electron microscopy. Hardness testing was performed using the Vickers technique.

A description of the methodology is given and the results of an analysis of two low alloy weld metals are reported for the purpose of demonstration. Through the demonstration it becomes clear that the two central parts of the methodology, microstructural characterisation and thermal analysis, in combination, offer the information needed to produce reliable CCT-diagrams.
PREFACE

This final thesis reports results from the work performed at ESAB AB during the period from October 2005 to February 2006, under the supervision of Dr. Mattias Thuvander at ESAB and Prof. Magnus Odén at Luleå University of Technology (LTU). The thesis is a part of my degree in Engineering Physics at LTU.
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1 INTRODUCTION

1.1 Background

The mechanical properties of a welded joint are related to the joint’s microstructure which, in turn, depends on its phase transformation history. Chemical composition and cooling rate are factors influencing the nature of the phase transformations and the temperatures at which they occur. Such information is often visualised with a continuous cooling transformation (CCT) diagram and can be used to predict the microstructural evolution of a metal after solidification.

![Typical CCT-diagram](image)

Figure 1-1 Typical CCT-diagram, showing cooling curves superimposed on the different phase areas. The diagram is read along the cooling curves from upper left to lower right [1].

In order to obtain favourable and stable properties of a welding material it is useful to acquire knowledge of its transformation behaviour within a certain range of cooling rates. The cooling rate is, in welding, commonly described by the cooling time from 800 to 500 °C, a temperature interval through which temperature often decreases linearly. This quantity, called $\Delta t_{8-5}$, is for welding often too small to be replicated with traditional analytical methods such as dilatometry. Along with other inadequacies described in the following section, it is apparent that other methods of analysis are called for. Direct, in situ, measurements of phase transformation related quantities are obviously better in terms of replicating actual welding conditions. Temperature is a quantity suitable for in situ measurements due to the relative simplicity of the experimental setup and the exothermal nature of phase transformations.
## 1.2 Alternative methods

The first CCT-diagrams were constructed by end quenching Jominy bars and studying the microstructure at various distances from the quenched end of the bar. Since the cooling rate depends on this distance several cross-sections of the bar can be studied, each corresponding to a certain cooling rate. Studying the micro constituents and calculating the cooling rate at various locations of the bar, provides the information necessary to construct a CCT-diagram.

Later, the dilatometer has become an important instrument for the development of CCT-diagrams. For a constant cooling rate the dilatometer is able to detect length changes of the test sample associated with phase transformations. By subjecting a number of specimens to different cooling rates and logging the transformation temperatures, transformation curves can be plotted to form a CCT-diagram. Microstructural characterisation of the samples determines which curve corresponds to which phase transformation [1].

The two methods mentioned above are of an analytical kind and usually lack the ability to replicate the thermal cycle of the weld. The transformation behaviour indicated by, for example, dilatometry on a test sample, might be quite different from that exhibited by the metal during actual welding conditions. Mechanical stresses and steep temperature gradients within the material are characteristic features of most welding processes and are known to affect transformation temperatures. One way of achieving better transformation data is to study the weld thermal cycle by direct, in situ, methods. These include, for example, thermal expansion measurements or, more commonly, thermal analysis for which a number of methods have been developed.

The phase transformations are exothermal in nature and therefore their effect on thermal histories can be registered by a thermocouple. When plotting the thermal data, phase transformations appear as small deviations from the otherwise smooth cooling curve as demonstrated in figure 1-2.

![Cooling curve from in situ measurements of a low-alloy weld metal. The curve shows a distinct bump where a transformation occurs.](image)

In principle, the methods of thermal analysis seek to locate phase transformations by identification of their exothermal effect. One way of doing this is by differentiating the
cooling curve, a process through which transformation effects are amplified, as well as any recorded noise. Amplification of noise is clearly unfavourable and can be avoided with the two thermocouple method developed by Phillip. The use of two thermocouples at the appropriate spacing provides both the cooling curve to analyse and a reference curve. Calculating the difference between the curves as a function of temperature makes transformations detectable without amplifying the noise [2]. The relative complexity of this technique and the fact that it is not applicable for measurements in the fusion zone have directed the development towards finding an alternative reference thermal cycle. Alternatives have been proposed where the reference cycle is obtained from theoretical models of the weld thermal cycle. A reference curve is then generated by numerically fitting a function, based on a heat flow equation, to the measured thermal cycle. An example can be seen in figure 1-3 [3,4,5].

Figure 1-3 A theoretical weld thermal cycle and definitions of important parameters. Time scale in seconds and temperature scale in degrees Celsius. The temperature refers to a point passed by an arc travelling at constant speed.

1.3 Aim
The aim of this work is to develop a methodology and a set of tools for the construction of in situ CCT-diagrams based on differential analysis of weld thermal histories.
2 WELDING METALLURGY

2.1 Phase transformations

Physically, an alloy is simply a system that consists of a mixture of phases. A phase can be defined as; “a portion of the system whose properties and composition are homogeneous and which is physically distinct from other parts of the system” [6]. Transformations between such phases are the result of the system’s desire to increase its stability, trying to reach an equilibrium state. Classical thermodynamics states that a system, at constant pressure and temperature, will be in equilibrium when its Gibbs free energy G, has a global minimum value. Apart from these stable states, there also exist metastable equilibrium states at local minima of the Gibbs free energy. During a phase transformation in, for example, a weld bead subjected to a certain degree of undercooling, the system decreases its free energy. This energy will be released as heat which will increase the temperature of the system and if the amount of the transformed phase is sufficient, the heat addition will be measurable [6].

In single component systems all phases have the same composition making the free energy dependent only upon temperature and pressure, whereas in multiple component systems, for example alloys, it has a composition dependence. This is the basis of the phase diagram. In addition to the basic components of a binary system such as iron-carbon in steels, a number of alloying elements can be added to control the stability of different phases. Generally these are austenite and ferrite stabilizers which are used to control the decomposition rate of austenite. In a CCT-diagram, a more stable austenite phase will have the effect of moving the transformation curves to longer times and lower temperatures.

2.2 Microstructure

2.2.1 Ferrite

Several types of ferritic structures may be encountered when studying the microstructure of weld metals. These include allotriomorphic ferrite $\alpha$, Widmanstätten ferrite $\alpha_w$ and acicular ferrite $\alpha_a$. The appearance of each phase may differ considerably depending on carbon and alloying content as well as cooling rate.

Allotriomorphic ferrite grows from the austenite grain boundaries and can sometimes be seen as almost continuous grain boundary layers of various thicknesses. Acicular ferrite on the other hand, nucleates inside the grains with transformation mechanisms similar to those of bainite. Widmanstätten ferrite nucleates and grows from the $\alpha$ layers and progresses into the austenite grains in the form of thin wedge shaped plates. The $\alpha_w$ plates grow fast and may within fractions of a second cross an entire grain. This is more common at higher cooling rates since the presence of acicular ferrite impedes the Widmanstätten progression for slower coolings [7,8,9,10].
2.2.2 Bainite

Bainite, which is a mixture of ferrite and cementite, forms when the cooling rate is too low for martensite to form, yet too high for ferrite to grow freely. When the austenite cools and ferrite starts to nucleate the carbon will not have enough time to diffuse into the austenite where its solubility is greater. The carbon diffuses to the grain boundaries and forms cementite when the concentration becomes sufficient. The bainite microstructure can vary considerably depending on at which temperature it forms. Upper bainite, formed at higher temperatures, has a lath-like structure consisting of alternate layers of cementite and ferrite. At the temperatures where lower bainite forms, the structure is finer and more plate-like with carbide precipitates often present, also inside the ferrite grains [6,8,11,12,13].

2.2.3 Martensite

At sufficiently high cooling rates the austenite will transform into martensite by a diffusionless shear process. The result is a hard and brittle lath-like microstructure. Since there is no time for carbon diffusion, the martensite will have the same composition as the austenite prior to transformation. During the martensite reaction the crystal structure changes into a body-centred tetragonal (B.C.T). The decomposition of austenite at these cooling rates is really a transformation into B.C.C ferrite, but due to the super-saturation of carbon, it is forced into a tetragonal structure. Often, some austenite is not transformed and this is termed retained austenite. It is also known that, for a steel of any given composition, the transformation temperature of martensite is essentially independent of cooling rate. However, at cooling rates low enough to produce bainite the martensite start temperature is suppressed slightly, as can be seen in figure 1-1 [6,14].

2.3 Welding processes and parameters

Arc welding processes have some basic similarities. For example, they all have some kind of power source providing the current and voltage necessary to produce the arc, a high current discharge, between the electrode and the work-piece. The arc forms a high temperature plasma that melts part of the work-piece creating a weld pool. Simultaneously, the plasma can be used to melt and transport filler material to the weld pool.

The weld consists of three important metallurgical zones; the fusion zone, the heat affected zone (HAZ) and the unaffected base material. The size, shape and properties of the fusion zone and the HAZ are influenced significantly by the welding process and the chosen welding parameters [15]. The welding parameters mainly affect the cooling rate of the weld, which is related to its final microstructure. That relation can be illustrated with the superposition of cooling curves on a CCT-diagram, as previously discussed in section 1.1.

There are three welding parameters of special significance to this work; heat input, preheat and interpass temperature. The latter two are of equal importance since they both are a means of controlling the rate at which heat flows from the heat source into the surrounding metal. Preheat is the temperature of the work-piece prior to the start of welding and interpass temperature is the temperature between adjacent runs of a multiple run weld. Heat input is the energy transferred from the power source per unit length of weld, which means that it also incorporates the influence of weld speed. The power used when welding with a particular process might not generate the same heat input when using another process since arc
efficiency can vary considerably between such techniques as shielded metal arc welding and gas tungsten arc welding.

2.3.1 Shielded metal arc welding (SMAW)
Shielded metal arc welding, sometimes referred to as manual metal arc (MMA) welding is the oldest arc welding method. It uses a coated metal electrode through which a current is passed. The primary functions of the coating are to improve the stability of the arc, generate a shielding gas protecting the molten metal and adding alloying elements. In SMAW, the electrode also serves as filler material.

![Figure 2-1 Schematic diagram of SMAW [16].](image)

2.3.2 Gas tungsten arc welding (GTAW)
Gas tungsten arc welding, also known as the tungsten inert gas (TIG) method, uses a tungsten electrode as source of the arc. If filler material is needed, it is added separately to the weld pool. Since no coated electrodes are used, as in the case of SMAW, the arc and weld pool are protected by an inert gas flow. Ignition of the arc is achieved either by bringing the electrode in contact with the work piece or by ionizing the gas between the electrode and work-piece with a high frequency discharge.

![Figure 2-2 Schematic diagram of GTAW [16].](image)
2.4 Modelling the heat flow

To mathematically describe the cooling of a weld the differential equation of heat flow can be used as a starting point.

\[
\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} = 2\lambda \frac{\partial T}{\partial t}
\]  

(1)

The thermal conductivity \( \lambda \), of the base material, is assumed constant. Of course, this assumption is incorrect but necessary to make the equation easier to solve. The solution,

\[ T(t) = a_1 + \frac{a_2}{\sqrt{t}} \cdot \exp \left( \frac{a_3}{t} \right) \]

(2)

is called the Rosenthal solution and shows the expected temperature at time \( t \) for a point passed by a moving heat source.

However, (2) is valid only for 2-dimensional heat flow, i.e. thin plates. For thick plates, the heat flow will be essentially 3-dimensional and the equation will take a slightly different form:

\[ T(t) = b_1 + \frac{b_2}{t} \cdot \exp \left( \frac{b_3}{t} \right) \]

(3)

The parameters \( b_{1,2,3} \) are directly related to the initial and peak temperature as well as the cooling rate and various material specific constants. The exact way in which they relate to one another is only of interest when estimating start values for the numerical fit described in the next chapter.

The critical thickness \( d' \)

\[ d' = \left\{ \frac{q}{v \rho c} \left( \frac{1}{1073 + T_0} \right) + \left( \frac{1}{1073 + T_0} \right)^{\frac{1}{2}} \right\} \]

(4)

defines the boundary between 2- and 3-dimensional flow and is a function of the initial temperature \( T_0 \), heat flux \( q \), weld velocity \( v \) and specific heat per unit volume \( \rho c \) [17].

Figure 2-3 The heat flow directions for 3-dimensional (left) and 2-dimensional (right) flow. The difference in plate thickness is related to the heat input.
3 THERMAL ANALYSIS

It can be difficult, if at all possible, to visually distinguish a phase transformation’s start and finish temperatures, from the recorded thermal history. If a cooling curve can be constructed from a theoretical model to fit experimental data fairly well, it can be used as a reference to make exothermal effects more easily detectable. The magnitude of the difference between experimental and reference temperatures makes it possible to locate phase transformations. For the purpose of faster and easier thermal analysis, some Scilab functions were created to facilitate loading, plotting and analysis of test data [18].

The thermal data acquired from in-situ measurements of the cooling weld metal is processed in the Scilab functions in the following main steps:

- Selection of curve sections for analysis.
- Fitting a mathematical model to experimental data by a least square parameter identification.
- Selection of transformation temperatures.
- Plotting of selected transformation temperatures superimposed on the cooling curves, creating a semi-logarithmic CCT-diagram.

3.1 Mathematical model

A good reference curve is one that is approximately identical to the measured curve everywhere except at the locations of the phase transformations. That is to say, a curve that without exothermal effects describes the cooling of the weld. To construct such a reference curve a mathematical description of the temperature as a function of time is needed. Since the experiments throughout this work have been performed under 3-dimensional conditions, an appropriate model can be the Rosenthal equation (3) presented in section 2.4.

When fitting the acquired data to (3), one important parameter is left out; a point of reference where \( t=0 \). Since only the cooling part of the thermal cycle is recorded by the data acquisition (DAQ) system, there is no way of knowing the time \( t_0 \) of the initial rise in temperature, as illustrated in figure 3-1. Including this parameter in the model function results in a new expression:

\[
T(t) = b_1 + \frac{b_2}{t-t_0} \cdot \exp\left(\frac{b_3}{t-t_0}\right)
\]  

Using (5) in the least square fit causes a very unstable behaviour, most likely due to the competitive influence of \( b_2 \), \( b_3 \) and \( t_0 \). Consequently it was found more suitable to iterate through different values of \( t_0 \). For each iteration a new fit of parameters \( b_1 \), \( b_2 \) and \( b_3 \) is performed, aiming at a specific shape of the difference (D) curve.
3.1.1 Model limitations

The Rosenthal solution of the heat flow equation is based on some rather rough approximations whose effects are not negligible when analysing the entire weld cycle. The constant conductivity approximation is inaccurate in more than one way. It is temperature dependent and can vary significantly from one phase to another in iron-based alloys. Furthermore, heat flow is never strictly three-dimensional or two-dimensional. It is often something in between, gradually changing during the cooling cycle in a way dependent on properties like plate dimensions and heat input. At higher temperatures the cooling rate may also depend to a large extent on heat radiation from the surface of the work piece, an effect not accounted for in the model.

The Rosenthal equation gives a satisfactory description of the cooling of the weld down until the initial decomposition of austenite at $T_{\text{start}}$. That means that above $T_{\text{start}}$ a good curve fit can be obtained. The case is not the same for lower temperatures. All temperature measurements made throughout this work show that cooling is more rapid after a transformation than it was before. This behaviour could be explained by a changing temperature distribution in the material during the transformation. The latent heat released through the transformation produces a local decrease in the cooling rate at the point of measurement. However, the surrounding metal, not undergoing transformation, continues to cool at approximately the same rate. As a result, the temperature gradients around the fusion zone will be steeper, and the cooling rate higher, at the time of transformation finish.

These weaknesses of the Rosenthal solution complicate curve fitting and make it hard to get good accuracy over large temperature ranges. To bypass some of the problems described above the cooling curve can be evaluated piecewise, hence limiting the influence of temperature or time dependent parameters not accounted for in the model.
3.1.2 Analysis of large temperature intervals

This section is based on the work of Alexandrov and Lippold about which more information can be found in references [3] and [4].

An “optimal fit” is equivalent to a minimal sum of squared errors and need not be the best fit for the purpose of finding phase transformations. Unless of course the optimal fit is ideal, i.e. identical to the cooling curve, a case not likely to occur in reality.

The reason why the optimal fit can be unsuitable lies in the definition of the difference function $D(T)$:

$$D(T) = T - T_R$$

where $T$ is the actual, or measured, temperature and $T_R$ is the reference temperature.

If the curve of optimal fit is used as reference, experimental values are likely to be located both above and below the reference curve, primarily because of inaccuracies in the model. The result will be a complicated appearance of the function $D(T)$. It is more desirable for $D(T)$ to behave in a predictable and constant manner in the absence of transformations. For instance, if

$$D(T) = k \cdot T$$

any phase transformations would appear as steps on an otherwise straight line in a $D$ versus $T$ diagram. In practice this is done by introducing the factor $k$, which determines how much the reference curve is to deviate from the optimal fit. Each point on the optimal curve is then displaced by an amount proportional to its derivative.

$$T_R = T_{opt} - k \cdot \frac{dT_{opt}}{dt}$$

This procedure effectively changes the derivative of the reference curve and produces a $D(T)$-curve more similar to the one described by (7). At the same time, the accuracy of the indicated transformation temperatures changes since they are defined as the temperatures of equal time-derivatives. That means,

$$\frac{dT}{dt} = \frac{dT_R}{dt}$$

at the time of start and finish of the transformation. Also, if the deviation is too great, the relative thermal contribution of a phase transformation will be too small to locate on the $D(T)$-curve. The “resolution” could be said to decrease.

As an effect of the deliberately reduced accuracy and the inherent lacks in the model for large temperature spans, this method of curve fit analysis is not ideal. As a result, the use of this method should therefore be limited to determining the approximate temperatures of transformations. Once that information is obtained a more thorough analysis can be performed.
3.1.3 Analysis of smaller temperature intervals

Assume that, for a given thermal history, the approximate start temperature for austenite decomposition is known. Then it is possible to choose a section of the curve of appropriate size in the neighbourhood of that temperature. This allows for a good fit over that section and the subsequent extrapolation of values into the transformation region. A typical appearance of the resulting D(T)-curve can be seen in figure 3-2. The difficulty in determining exactly where the D-curve starts to deflect is one reason why the transformation start is defined in the way depicted in figure 3-2. Another reason is that this definition decreases the influence of the quality of the least square fit. Local changes in the derivative of D(T) can also be seen, indicating that several transformations have occurred. In general,

\[
\frac{d^2 D}{dT^2} < 0
\]  

(10)

when a transformation accelerates and

\[
\frac{d^2 D}{dT^2} > 0
\]  

(11)

when the transformation decelerates and its thermal contribution vanishes. The comparatively small size of these thermal effects demands a separate curve fit of that area in order to determine their locations more accurately. Nevertheless, just noting their presence is useful since it indicates that a mixed microstructure is to be expected.

![Figure 3-2](image)

**Figure 3-2** Left: Typical appearance of a difference curve. Right: Diagram showing how the measured curve is divided into sections for more accurate analysis.
4 EXPERIMENTAL DETAILS

4.1 Production of the weld metal

The weld metal was deposited to produce a bead-on-plate using SMAW. Multiple runs were made to minimize mixing of filler and base material, which could affect transformation temperatures.

The experiments presented in this work comprise the two filler materials OK 75.75 and OK 74.70. These materials were chosen because they undergo different phase transformations within the range of achievable cooling rates.

Other filler materials, with various amounts of alloying elements were also measured on but used primarily as test data during the process of methodology development.

Table 1. Weld metal composition (wt%)

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>S</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>OK 74.70</td>
<td>0.08</td>
<td>0.4</td>
<td>1.5</td>
<td>-</td>
<td>-</td>
<td>0.4</td>
</tr>
<tr>
<td>OK 75.75</td>
<td>0.06</td>
<td>0.3</td>
<td>1.6</td>
<td>0.35</td>
<td>1.7</td>
<td>0.4</td>
</tr>
</tbody>
</table>

4.2 Temperature measurements

Part of the weld metal was re-melted using a GTAW torch and the thermocouple manually plunged into the weld pool. The welding current was set at different values between 200 and 300 Amperes, higher currents giving slower cooling rates. Thermal data was recorded during a period long enough for all expected transformations to occur.

To obtain reliable data it is important to ensure that the depth of the weld pool is sufficient. My experiments show that less electromagnetic noise is recorded when the entire exposed part of the thermocouple is positioned inside the weld pool. In addition, it was also found desirable to avoid contact between the thermocouple and the solid metal beneath the weld pool.

4.2.1 Data acquisition system

The temperature was measured using a S-type thermocouple (Pt-Pt/Rh) while the data was logged with a Dewetron DAQN-THERM module. This configuration allowed temperatures in the range from 0 to 1750°C to be recorded. The system was sampled at 1000 Hz by the LabView software running on an electromagnetically shielded computer. A schematic diagram of the setup is shown in figure 4.1.
Figure 4-1 Schematic diagram of the experimental setup for temperature measurements in the fusion zone.

Figure 4-2 Left: Picture of the experimental setup, a) Shielded computer, b) Shielded DAQ-module, c) Thermocouple, weld metal and water bath. Right: Thermocouple positioned in the solidified weld metal.

The thermocouple uses a thermoelectric property, called the Seebeck effect, and consists of two wires of dissimilar metals joined at their ends. Heating this junction produces a voltage across it that is a function of temperature and the composition of the two metals. By measuring this voltage, the corresponding temperature can be calculated. When preparing the two wires it needs to be ensured that connection and heating of the wires are limited to the endpoint of the thermocouple. Threading the wires through separate holes in an aluminium oxide cylinder, as shown in figure 4-3, ensures both thermal and electrical isolation.

All connections between wires of dissimilar metals in a circuit exhibit this thermoelectric behaviour but need not influence the measurement. For example, the thermocouple wires are connected to the DAQ-module through copper leads. In this case the connections between platinum and copper all generate a small voltage but the sum of their contribution is zero. This is called the intermediate metal law and it states that a third metal inserted between the
dissimilar metals of the thermocouple does not affect the thermoelectric voltage of the circuit. It applies to the case when the two extra junctions, created by the insertion of the third metal, are at the same temperature [19].

![Figure 4-3](image1.jpg)

**Figure 4-3** Typical thermocouple configuration for measurements in the fusion zone.

### 4.2.2 Control of the cooling rate

Since transformation start and finish temperatures depend on cooling rate each material was preheated to different temperatures between room temperature and approximately 400 °C. In this way the heat flow and consequently the cooling rate at the point of measurement could be altered. To achieve cooling rates faster than that of a plate at room temperature, experiments were done with forced cooling where the backside of the plate was submerged in water. The cooling rate can also be altered by changing the heat input or the dimensions of the work piece.

![Figure 4-4](image2.jpg)

**Figure 4-4** Parameters with which cooling rate can be altered.

### 4.3 Microstructural characterisation

Once the temperatures of the phase transformations have been determined, their nature also has to be characterised before a CCT-diagram can be completed. During this procedure, the microstructure developed for different cooling rates is studied and by estimating the amount of the constituents present conclusions can be drawn about the transformations experienced by the material.
4.3.1 Specimen preparation
In order to reveal the microstructure of a sample it needs to be polished and etched. The etchant attacks microconstituents with different degrees of aggression giving rise to topographic contrast in the microscope.

The samples investigated were cut out of the weld with one surface as close to the position of the thermocouple as possible, shown in figure 4-5. They were then mounted in bakelite and polished with diamond solution of decreasing particle size down to 1µm. The samples were then etched using 1% Nital.

![Figure 4-5 Illustration of where the microscopy specimen is cut from the weld metal.](image)

4.3.2 Light optical microscopy (LOM)
LOM is perhaps the most common method for microstructural characterisation of weld metals. It can be used to identify many of the microconstituents present in a weld metal specimen. Image contrast is primarily the result of the surface roughness which causes light to be irregularly reflected. Contrast can also, to some degree, be a result of the light absorption properties of the different phases. LOM has two major limitations; depth of field and resolution, the latter of which makes identification of small constituents impossible [20].

The light optical micrographs in this work were produced with a Leitz Aristomet microscope.

4.3.3 Scanning electron microscopy (SEM)
Scanning electron microscopy can be used to produce topographic images of much higher resolution than LOM images. The basic structure of a SEM consists of an electron source, a column of electrostatic or magnetic lenses for control and focus of the electron beam and detectors for the various signals generated at the sample. The electron source is often a resistance heated filament from which thermionic electrons are emitted and accelerated. A field emission gun (FEG) constitutes a more pointlike and intense electron source, with which better imaging properties can be obtained.

Electron microscopy was performed with a Leo Ultra 55 FEGSEM in the secondary electron mode. Secondary electrons are generated when the electron beam knocks electrons out of the conduction band of the sample. These are of such low energy that only the ones generated
very close to the surface are able to leave the material. This makes secondary electrons suitable for imaging of high topographic contrast [21].

4.3.4 Hardness testing

Hardness is a measure of a material’s ability to resist plastic deformation. The Vickers hardness test consists of pressing a pyramid shaped diamond into the surface of the sample. Measurements of the indentations are then performed with the help of a microscope and the hardness value is calculated from the definition below.

\[
HV = 1.854 \cdot \frac{P}{d}
\]

In (11) \(P\) is given in kilograms and \(d\) in millimetres.

Microhardness was measured with the Vickers technique. A load \(P\), of 5 kg was applied at a number of equally spaced points across the fusion zone. The indentations were made on a line parallel to the top surface of the weld bead.
5 RESULTS

The methodology described in the previous chapters can be summarized schematically as in figure 5-1.

In the following sections of this chapter the results of the application of this methodology will be presented. The results are to be seen as a demonstration of the method and not considered a complete CCT analysis. The microscopy images have been chosen to be representative of the sample as a whole. First, a number of LOM images will be presented and in the cases where it was thought to be of interest, FEGSEM images have been added. The higher resolution of the FEGSEM is necessary to distinguish between lower and upper bainite and detect the presence or confirm the absence of retained austenite.

The values of $\Delta t_{8.5}$ are calculated automatically by the Scilab functions and given with two decimals. The accuracy is dependent on the relation between cooling rate and sampling frequency. In the following cases the sampling rate after mean value reduction is 100 Hz which is too low to justify the use of hundredths of a second for $\Delta t_{8.5}$. However, for the sake of consistency between the CCT-diagrams and the results from microanalysis, hundredths will be used throughout the entire chapter.
5.1 Light optical microscopy

**Figure 5-2 OK 74.70, Δt₈₋₅ = 0.94 s**

The light optical micrographs show a structure composed mainly of martensite.

**Figure 5-3 OK 74.70, Δt₈₋₅ = 4.36 s**

When the cooling rate is reduced the microstructure has turned bainitic.

**Figure 5-4 OK 74.70, Δt₈₋₅ = 12.03 s**

Further reduction of the cooling rate gives an increased grain size but still a bainitic microstructure.
At this high cooling rate the microstructure consists of bainite and small amounts of martensite.

This microstructure is, in the LOM, very similar to the one in figure 5-5.

This sample consists of tempered bainite which is a result of the high preheat temperature used to achieve the slow cooling.
5.2 Scanning electron microscopy

![Scanning electron micrographs, OK 74.70, Δt_{8.5} = 0.94 s.](image)

At this magnification it is clear that the fastest cooled sample consist not only of martensite, but also of small amounts of lower bainite. In the upper micrograph lower bainite is marked B and martensite M. In the lower micrograph there is no clear indication of the martensite laths being separated by retained austenite. The white lines are likely due to the higher intensity of secondary electrons emitted as a result of those areas higher topography.
The sample subjected to intermediate cooling can be seen to consist of a mixture of upper bainite (uB) and lower bainite (lB). The carbide precipitates characteristic of lower bainite are clearly resolved and can be seen as white, closely packed dots inside the grains. The larger grains of darker contrast are upper bainite.
For the slowest cooling ($\Delta t_{8-5} = 12.03$ s) there is no lower bainite present and the grains have grown much larger. As can be seen in the images above, upper bainite is dominating the microstructure. Thin plates of cementite, separating adjacent layers of ferrite, can be seen as white lines in the diagonal direction of the lower image.

Figure 5-10 Scanning electron micrographs, OK 74.70, $\Delta t_{8-5} = 12.03$ s.
5.3 Thermal analysis

In this section the results from the least square fit and the differential analysis will be presented in the form of difference curves.

Figure 5-11 OK 74.70, $\Delta t_{8,5}$=12.03 s

Figure 5-12 OK 74.70, $\Delta t_{8,5}$=4.36 s
In situ Detection and Characterisation of Phase Transformations in Weld Metals

Figure 5-13 OK 74.70, Δt_{8.5}=0.94 s
5 Results

Figure 5-14 OK 75.75, $\Delta t_{8,5} = 1.89$ s

Figure 5-15 OK 75.75, $\Delta t_{8,5} = 3.00$ s

Figure 5-16 OK 75.75, $\Delta t_{8,5} = 3.91$ s

Figure 5-17 OK 75.75, $\Delta t_{8,5} = 75.39$ s
5.4 Hardness

As could be expected the hardness decreases with the cooling rate, both as a result of the increased grain size and the mechanical properties of the different phases.

![Graph showing hardness vs. position](image)

**Figure 5-18** Results from Vickers hardness tests on OK 74.70. The distance between each indentation is approximately 1 mm. $\Delta t_{8,5}$ is displayed on the right hand side of the diagram.

![Graph showing hardness vs. position](image)

**Figure 5-19** Results from Vickers hardness test on OK 75.75. The distance between indentations is approximately 0.5 mm. $\Delta t_{8,5}$ is displayed on the right hand side of the diagram.
5.5 CCT-diagrams

Combining the result from the thermal analysis and the characterisation of the microstructure allows the diagrams in figure 5-20 and 5-21 to be constructed. To separate the different phase regions accurately, as in figure 1-1, more cooling curves need to be analysed. Linear interpolation between the marked points in the diagrams can give a rough estimate of the transformation temperatures for near lying cooling rates.

Since the martensite start temperature is known to be independent of cooling rate it can be concluded that faster cooling than $\Delta t_{8-5} = 0.94$ s will result in martensite transformations at the temperature indicated by $M_s$.

![CCT-diagram](image)

**Figure 5-20** The results from section 5.1 and 5.2 combined into a single diagram. The nature of the transformations is given by the notation $M_s$ (martensite start) and $B_s$ (bainite start).

In figure 5-21 it can be seen that more than one phase transformation occurs for the higher cooling rates. The LOM images show a bainitic structure and it is likely that the bainite transformation is followed by a martensite transformation at the temperature indicated by $X_s/B_f$ in figure 5-21. The tempered bainite obtained for the slowest cooled sample is a result of the high preheat temperature used. As the point of measurement approaches thermal equilibrium with the surrounding metal, the cooling rate of the weld is determined primarily by the rate at which heat is transferred to the surrounding air. Since that is governed by convection and radiation the cooling becomes very slow as the measured temperature approaches the preheat temperature. This is an unwanted effect since the interest lies in studying the CCT-behaviour of weld metal and not post weld heat treatment.
Figure 5-21 CCT-data from the analysis of OK 75.75. Bs indicates the bainite start temperature, Bf the bainite finish temperature and Xs the start of what is likely a martensite transformation.
6 CONCLUDING REMARKS

The combination of microstructural characterisation and thermal analysis outlined in this work can be used as a fast and easy way to map the transformation behaviour of weld metals. The experimental setup can be kept relatively simple and the measurements can be performed in situ, which is an advantage over dilatometry.

The cooling rate is easily controlled with the variation of the preheat temperature but the curve fitting becomes less accurate as the preheat temperature increases. Also, if it is too high, as seen in the slowest cooled OK 75.75 sample, it can have the effect of post weld heat treatment and the resulting microstructure will not be the same as the one produced under actual welding conditions.

The accuracy of the results is directly dependent on the construction of a high quality thermal reference cycle that can be based on the Rosenthal equation. However, it would be an advantage to use a better theoretical description of the weld thermal cycle.
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8 REFERENCES


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