Non-destructive in situ monitoring of the microstructural development in high performance steel components during heat treatment

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A new monitoring system was developed, that enables a contact-free and in situ, non-destructive monitoring of the material´s transformation and microstructure´s development during the cooling phase from the austenitic region. For this purpose characteristic changes in the electrical and magnetic properties that occur during the formation of ferromagnetic phases and microstructures are measured using eddy-current technology and the analysis of harmonic signal components. The monitoring system provides information about the onset of the transformation, the current transformation level and the end of the transformation in steel components as well as information about the evolution of the microstructural components, i.e. ferrite-pearlite, bainite and martensite. By evaluating the measured signal online in real time it is possible to classify and differentiate the evolution of microstructures, to regulate the cooling parameters, and therefore to specifically set the component's properties. The monitoring system and the robust eddy-current sensors can be employed in industrial environments at component temperatures of up to 1250 °C in different environments, such as salt-baths, furnaces, spraying fields or in air.

KEYWORDS: IN SITU MONITORING SYSTEM - PHASE TRANSFORMATION - MICROSTRUCTURE EVOLUTION - FORGING PROCESS - QUALITY ASSURANCE - EDDY-CURRENT TECHNOLOGY - HARMONIC ANALYSIS

INTRODUCTION

The shortage of resources and the continuously increasing importance of climate protection require high-performance lightweight components for machines and vehicles, as well as resource- and energy-efficient manufacturing methods. In order to meet these objectives, high strength and ductile bainitic steels are increasingly employed, which can be formed in a shortened forging process chain directly from the forging heat.

Especially in the forging processes with integrated heat treatment many process parameters, i.e. the cooling rate, the austenitising temperature and duration, the level of deformation and the local and global chemical compositions, have a drastic influence on the phase transformation and the evolution of the microstructure in the component´s surface region and core. Therefore making predictions about the material transformation during the cooling phase from the austenitic state based on CCT diagrams and simulated computations are only possible to a very limited extent.

Reliably setting of the required component properties directly from the forging heat is essential for high performance forging components and requires an individual adapted heat treatment. A monitoring system that provides information about the phase transformation and the resulting properties could be used for regulating the cooling process and also for an online quality assurance.

However, all the currently available methods for an in situ detection of phase transformation and microstructure evolution focus on laboratory applications for research purposes and are not suitable for use on components in industrial environments [1], [2], [3].

Within the framework of the AiF-Initiative "EcoForge", which has the objective of manufacturing resource- and energy-efficient high-performance components by means of controlling the cooling and specifically setting bainitic microstructures directly from the forging heat [4], [5], [6] an appropriate monitoring system was developed. The system provides information about the material´s transformation behaviour and the evolution of ferromagnetic phases and microstructures in geometrically complex forged steel components during their cooling from the austenitic region even in industrial environments [7].

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MEASURING PRINCIPLE AND TESTING SYSTEM

The measuring principle of the monitoring system is based on eddy-current technology and harmonic analysis of eddy-current signals in ferromagnetic materials. An alternating primary magnetic field is generated using an excitation coil subjected to a sinusoidal alternating current.

This magnetic field leads to a continuous magnetisation along the magnetic hysteresis curve in ferromagnetic materials. At the same time, eddy-currents are induced in the component. These currents produce a secondary magnetic field that will oppose the primary excitation field. The resulting magnetic field obtained in this way is non-linearly distorted according to the magnetic hysteresis and induces an alternating voltage with harmonic signal components in the measuring coil. These higher harmonics can be separated and evaluated via a fast Fourier transform and allow for a non-destructive characterisation of the mechanical material properties. Because of the good correlation between the measured electromagnetic values and the material´s physical properties the harmonic analysis of eddy-current signals is a reliable technology for material characterization [8], [9].

The newly developed monitoring system consists of a measuring and control PC for generating the excitation signal and for recording the measured signal, a power amplifier and the eddy-current sensor. The robust and high-temperature resistant sensors enable, for the first time, the harmonic analysis of eddy-current signals in steel components at temperatures of up to 1250 °C. Thus, information about the material´s transformation behaviour and the formation of ferromagnetic phases and microstructural components can be obtained online while cooling down from the austenitic state.

In the paramagnetic, austenitic state steel has a very low electrical conductivity and low magnetic permeability. Owing to the design of the eddy-current sensors, which are compensated to air, the amplitude of the 1st harmonic is approximately 0 V in this condition. Harmonic components in the measuring signal are based on the component´s magnetic properties and the magnetic hysteresis and occur only in ferromagnetic materials, so the amplitudes of the higher harmonic signal components also vanish.

During the phase transformation and formation of ferromagnetic microstructures the electrical and magnetic properties, i.e. the magnetic hysteresis curve, and the measuring signal change significantly, Fig. 1. The higher harmonics (here especially the 3rd harmonic) provide characteristic information about the magnetic properties and therefore information about the microstructure, while the 1st harmonic provides information about the magnetic and electrical properties. In earlier work, it was shown that, for the isothermal bainitic transformation occurring in a salt bath, a very good correlation exists between the 1st harmonic´s amplitude and the change in length recorded using a dilatometer, which corresponds to the degree of transformation [10], [11].

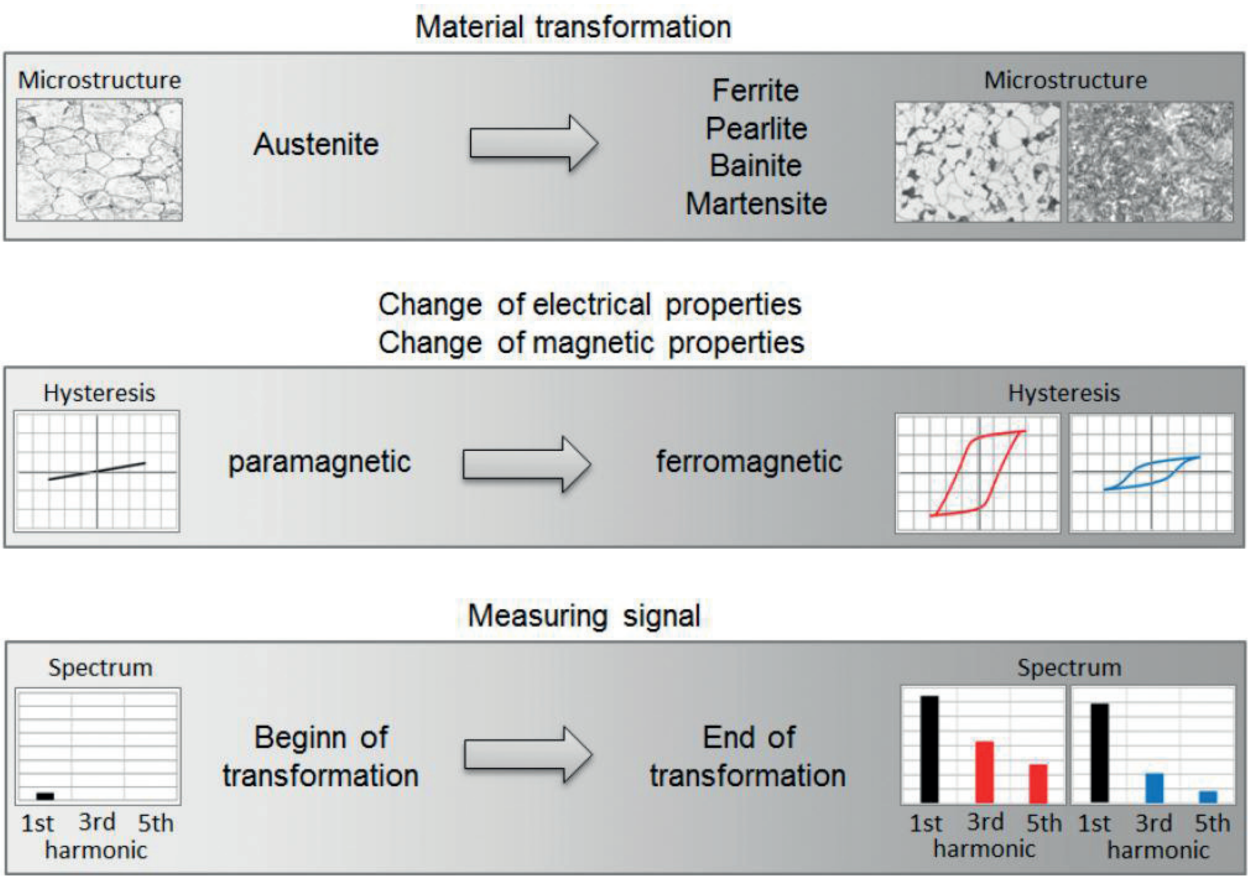

Fig. 1 - Measuring principle, see main text for details

MONITORING THE MATERIAL TRANSFORMATION UNDER LABORATORY CONDITIONS

In the following, the evolution of the characteristic signal of the 1st and 3rd harmonics during the phase transformation from the austenitic state will be discussed for the case of some cylindrical steel samples. The specimens, having dimensions of Ø15 mm x 80 mm, were austenitised in a chamber furnace and cooled down with a controllable fan (flow rate: 0…18 m/s). Temperature was measured using a thermocouple and the phase transformation was recorded using a water-cooled sensor with an internal diameter of 55 mm, Fig. 2.

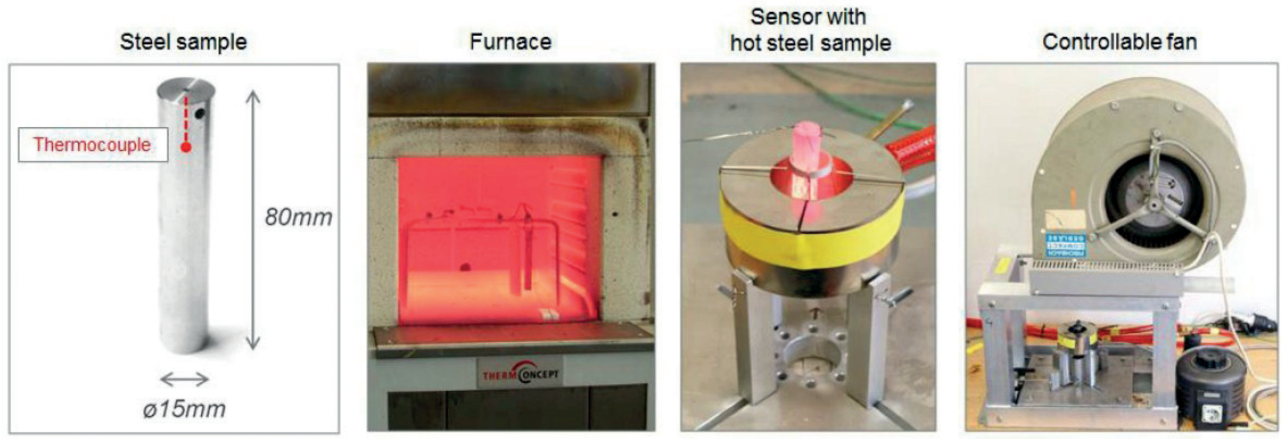

Fig. 2 - Experimental setup for cylindrical steel samples

1st HARMONIC

The 1st harmonic´s signal is influenced by both the changes in the electrical and magnetic material properties as a consequence of the formation of phases, i.e. the microstructural evolution, as well as the temperature dependency of the material properties. Fig. 3 shows the temperature-time-profile and Fig. 4 the 1st harmonic´s signal evolution for a cylindrical sample made of the case hardening steel 1.6587 (18CrNiMo7-6). The sample was austenitised at a temperature of 1200 °C for 25 minutes in neutral annealing coal (nc) in the chamber furnace and subsequently continuously cooled down in static air. During the cooling, slight scaling (mostly magnetite, Fe_3O_4) occurred on the surface. The temperature as well as the 1st harmonic´s signal is colour-coded in order to be able to chronologically map the evolution in the impedance plane.

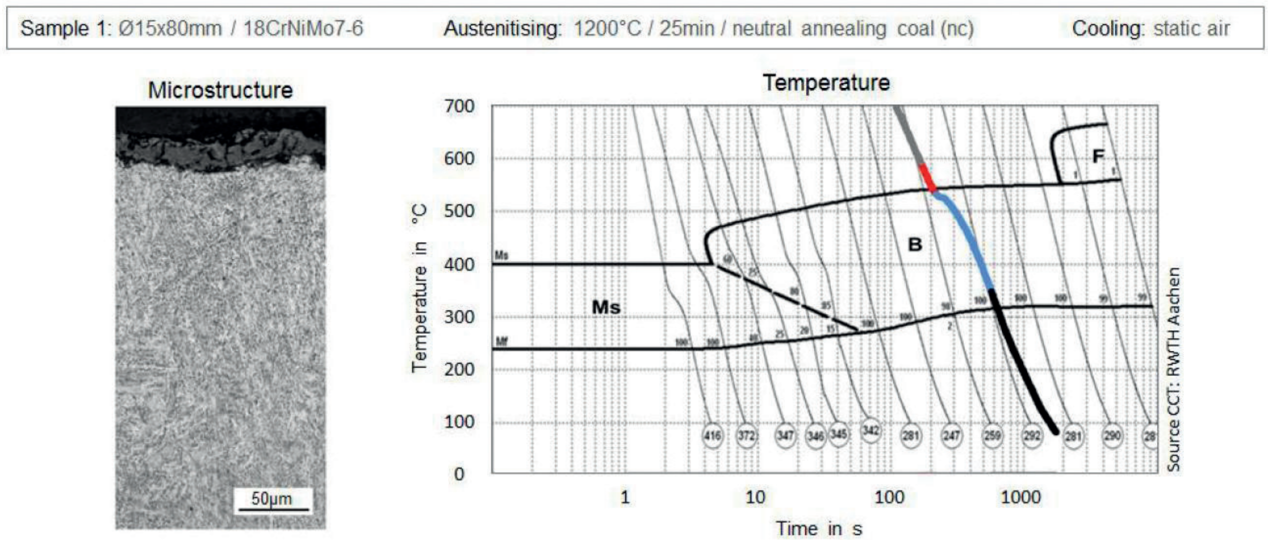

Fig. 3 - Optical micrograph of the bainitic microstructure along with scaling at the outer surface (left) and temperature-time-profile of sample 1 (right)

In the paramagnetic, austenitic state the amplitude of the 1st harmonic is approximately 0 V at the beginning of the measurement (grey curve). At the onset of cooling, no significant change occurs initially in the measured signal. After falling below the Curie temperature of magnetite (Fe_3O_4), at approx. 580 °C, ferrimagnetism sets in, which causes a slight rise in the amplitude (red curve).

During the subsequent bainitic transformation (blue curve), the 1st harmonic´s amplitude rises in proportion to the degree of the material transformation and reaches its maximum towards the completion of transformation. The actual degree of transformation can be estimated by the ratio of the 1st harmonic´s amplitude to its saturation amplitude at the end of phase transformation.

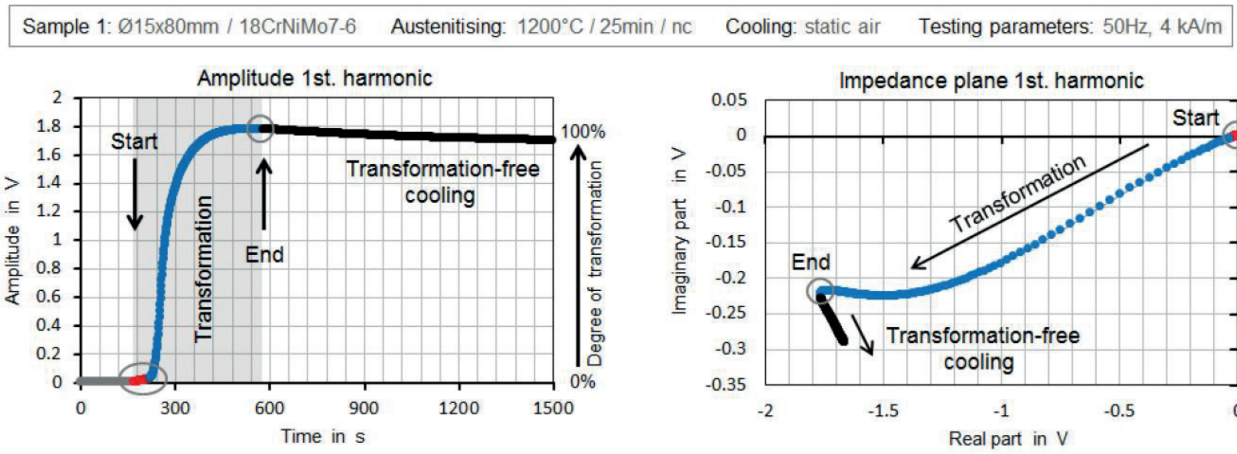

Fig. 4 - Evolution of the 1st harmonic for sample 1, which featured a bainitic microstructure with scaling after the transformation

After reaching the end of the transformation, a further transformation-free cooling of the sample (black curve) leads to a temperature-related change in the electrical and magnetic material properties and therefore to a slight decrease in the amplitude. The transition between the material transformation and the transformation-free cooling is characterised by an amplitude maximum in the 1st harmonic and by a significant change in the phase in the impedance plane. Thus, the microstructural transformation can be clearly differentiated from the transformation-free temperature change.

3rd HARMONIC

The measured signal´s higher harmonic components (3rd harmonic) occur due to the non-linear distortion of the measured signal as a consequence of the magnetization along the component´s magnetic hysteresis curve. Thus, in the paramagnetic, austenitic state, steel materials initially exhibit no significant magnetic hysteresis curve and no higher harmonic components in the measured signal. During the formation of ferromagnetic phases in the microstructure a characteristic change takes place in the shape of the magnetic hysteresis which leads to a characteristic change in the higher harmonic components. In the impedance plane, the 3rd harmonic now demonstrates as a characteristic loop. Its phase position in the impedance plane depends, to a large extent, on the microstructure and the corresponding properties and therefore allows for the characterisation of the phases and microstructures formed throughout the cooling from the austenitic state.

To separately detect the material transformations in the core and the surface region, the test frequency and field strength can be changed. For a test frequency of 50 Hz and a field strength of 4 kA/m, the penetration depth of the eddy currents and the volume which is probed are relatively large. Therefore the core region contributes much more than the surface region and the influence of surface scaling or decarburization to the signal can be effectively suppressed. By increasing the test frequency to 200 Hz and decreasing the field strength to 1.5 kA/m, the penetration depth and the volume which contributes can be significantly decreased and the technique becomes sensitive to the properties of the surface region. A change in the test frequency leads also to a significant change in the loop´s phase position in the impedance plane, so that the measuring results obtained at different frequencies are not directly comparable.

In addition to the temperature-time-path and the corresponding signal evolution of the 1st harmonic in Figs. 3 and 4, the 3rd harmonics for sample 1 are depicted in Fig. 5 for the 50 Hz and 200 Hz test frequencies.

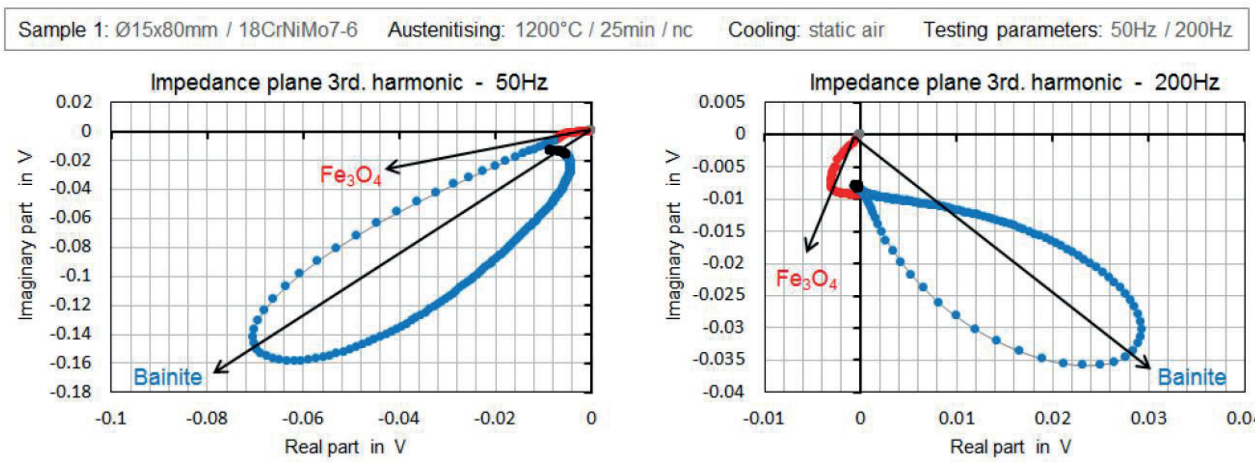

Fig. 5 - 3rd harmonic for sample 1 upon a transformation to a bainitic microstructure along with formation of scaling for two different test frequencies

In the austenitic state, no higher harmonics were initially detectable in the measured signal, i.e. the 3rd harmonic´s amplitude is approx. 0 V (grey curve) in Fig. 5. Upon falling below the Curie temperature of magnetite (Fe_3O_4) at approx. 580 °C (red curve), an initial rise of the 3rd harmonic´s amplitude occurs due to the formation of ferrimagnetic properties in the surface region. A first loop can be seen in the measuring signal at 200 Hz where the surface region's properties contribute for the overall signal. By contrast, the loop at 50 Hz is hardly visible. The bainitic transformation (blue curve) results in a second loop that can be seen at both frequencies and shows a considerable difference in the phase positions as compared to the first loop.

FERRITE-BAINITE

During the formation of mixed microstructures, the individual transformations, i.e. ferrite-pearlite, bainite and martensite, are frequently interrupted by regions where transformations are very slow. As an example, the measuring results of some ferritic-bainitic microstructure transformations are depicted in Fig. 6 for three 18CrNiMo7-6 cylindrical samples. The samples were austenitised in neutral annealing coal in the chamber furnace at temperatures in the range 850 °C to 925 °C and then very slowly cooled. Depending on the austenitising temperature the ferritic transformation is favoured or suppressed, and thus microstructures with different amounts of ferrite and bainite could be established. The detection of this strong influence of the austenitising temperature on the microstructural formation clearly shows the advantage of the monitoring system compared to the use of CCT diagrams or to simulations that are based on such diagrams.

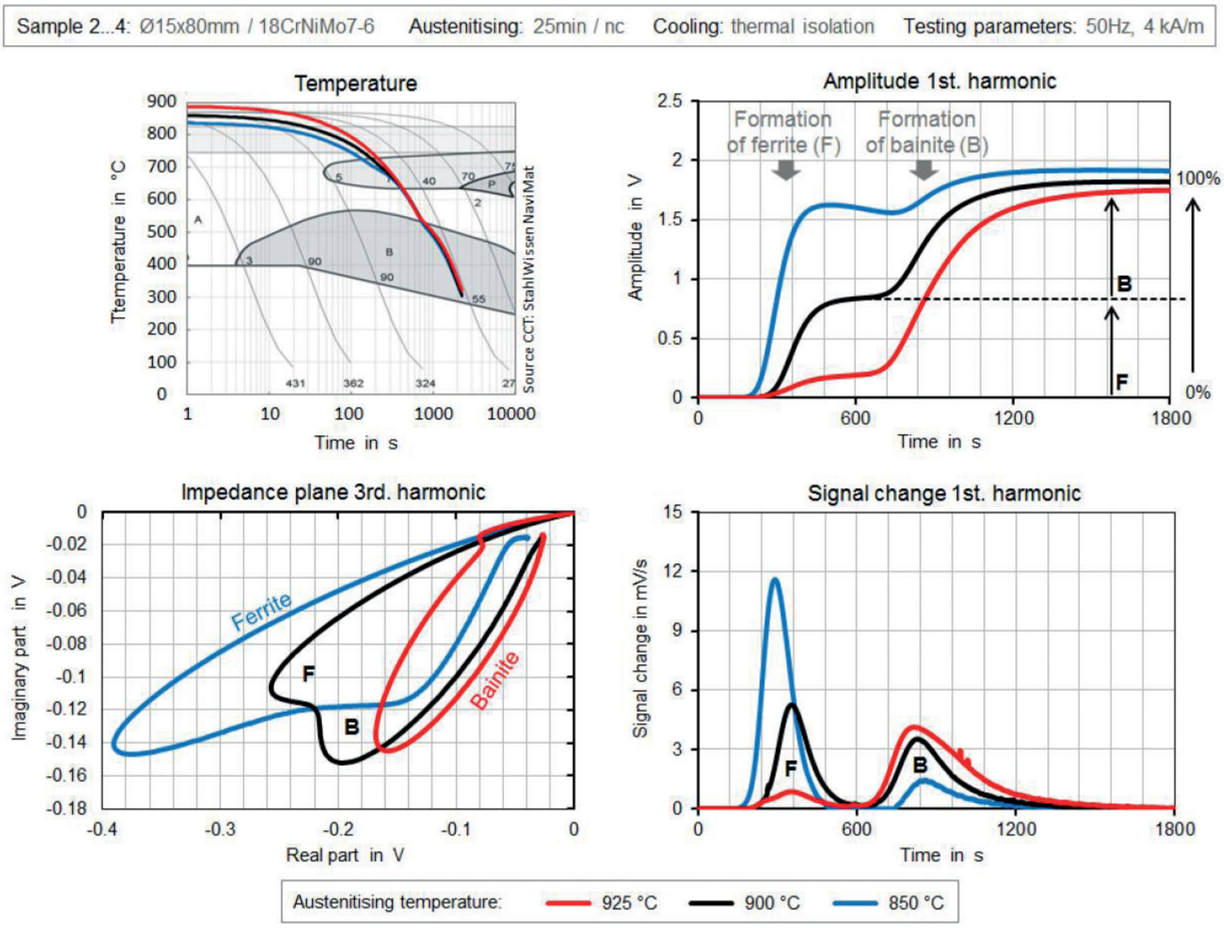

Fig. 6 - Temperature and signal courses of the samples 2, 3 and 4 with different ferritic-bainitic microstructures, partly recompiled from [7]

The amplitude of the 1st harmonic shows a large, slowly transforming region that separates the ferritic and bainitic transformation. Owing to the existing correlation between the amplitude of the 1st harmonic and the degree of transformation, a rough estimate of the ferritic and the bainitic fractions of the microstructure can already be made during the cooling phase. Another characteristic value that is very sensitive to changes in the transformation rate and therefore well suited for detecting phase transitions is the rate of the signal change of the 1st harmonics amplitude dV/dt.

By contrast, the 3rd harmonic´s course in the impedance plane, which shows two incomplete loops at considerably different phase angles, enables the development of the phases and microstructures to be characterized and the material transformations to be assigned to ferrite and bainite formation.

The microstructural composition, which was metallographically determined after the completion of the phase transformation, confirmed that the evolution of the mixed microstructure can be reliably recorded during the cooling phase and the fractions of the microstructure can be quantified, Fig. 7.

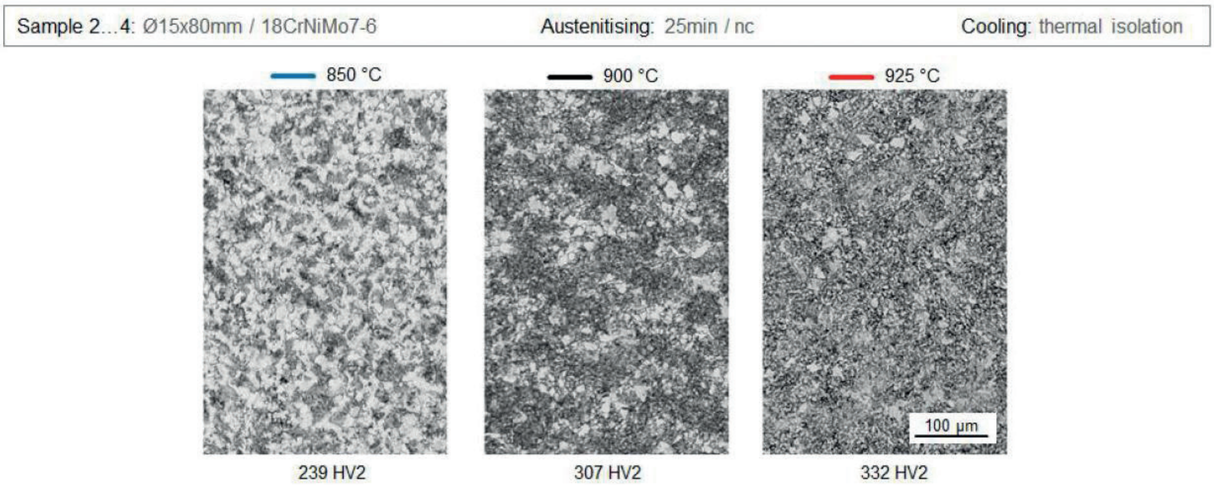

Fig. 7 - Optical micrographs of the microstructures formed in samples 2, 3 and 4

BAINITE-MARTENSITE

A mixed microstructure that consists of bainite and martensite is often difficult to characterize and to quantify by using conventional microscopy. Figure 8 depicts an example of a thin and fast cooled 1.7225 (42CrMo4) cylindrical specimen where the bainitic and martensitic transformation, also not shown in the CCT diagram, is separated by a slowly transforming region. This example shows that the characterisation and quantification of bainite and martensite can be much easier analysing the phase transformation during the cooling process than in the cold state. The signal of the 3rd harmonic shows three incomplete loops (the estimated full loop is marked by the dashed line): A slight ferrite formation at the beginning of the transformation, the bainite formation and the martensite formation are detectable for increasing phase angels. The analysis of the 1st harmonic reveals a microstructure that consists mainly of martensite with approx. 25% bainite and a negligible amount of ferrite. The corresponding temperature measurement shows a temperature-time-path that is somewhat lower than expected, due to the smaller sample diameter (10 mm), the high cooling rate under the fan and the exposed location of the thermocouple in the air flow at the top of the sample.

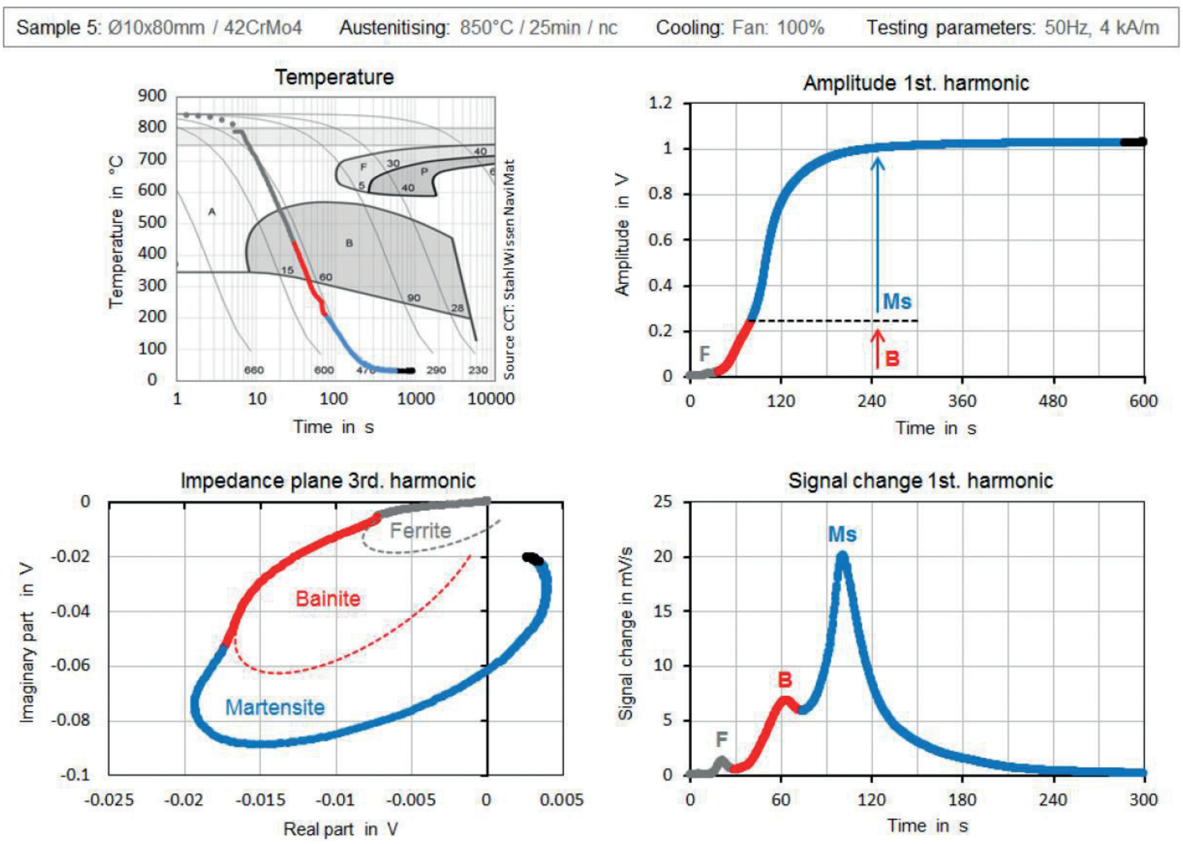

Fig. 8 - Temperature-time-path and evolution of the measured signal for sample 5 with a bainitic-martensitic microstructure

MONITORING THE PHASE TRANSFORMATION UNDER INDUSTRIAL CONDITIONS IN A FORGING LINE

In the following results from a test run with the monitoring system in a forging line under industrial conditions is shown. By recording the phase transformation and controlling the forging component´s cooling directly from the forging heat using a spray field, the applicability of the technology for applications in quality assurance and for setting the desired properties of a component were evaluated. The investigations were carried out on two demonstration components, a "stepped shaft" and a "common rail". Figure 9 shows the "stepped shaft", the position of the shaft in the sensor and the sensor with a hot sample in the water-air spray field.

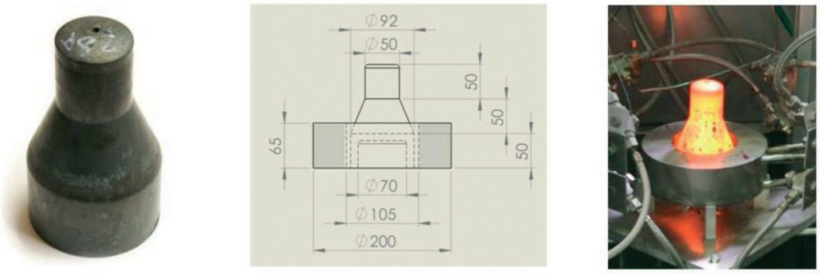

Fig. 9 - Demonstration component "stepped shaft" and experimental setup in the spray-field

CONTROLLED COOLING

The "stepped shaft" demonstration component was made of the high strength and ductile bainitic HDB-steel 22MnSiCr6-6-5. Here the objective was to rapidly cool the component to a temperature above the martensite start-temperature by employing the water-air spray field and then subsequently cooling in air to obtain a homogeneous bainitic microstructure in the lower bainite region. The geometry of the 150 mm long "stepped shaft", having a bulk conical chamfer at its middle and a thin-walled area at the lower end, was intentionally designed such that it represented a challenge for controlling the cooling in the spray field.

The results of the two-step controlled cooling of the "stepped shaft" in the water-air spray field and in static air are depicted in Fig. 10. Since no drilled hole was available for positioning a thermocouple during the forged component´s cooling from the forging heat, a thermocouple was unprotected pressed against the inner surface of the mid-section. For this reason, it was only possible to record a qualitative temperature measurement after the active cooling was finished. The expected temperature while cooling in the water-air-spray is marked by the dashed line in the CCT diagram.

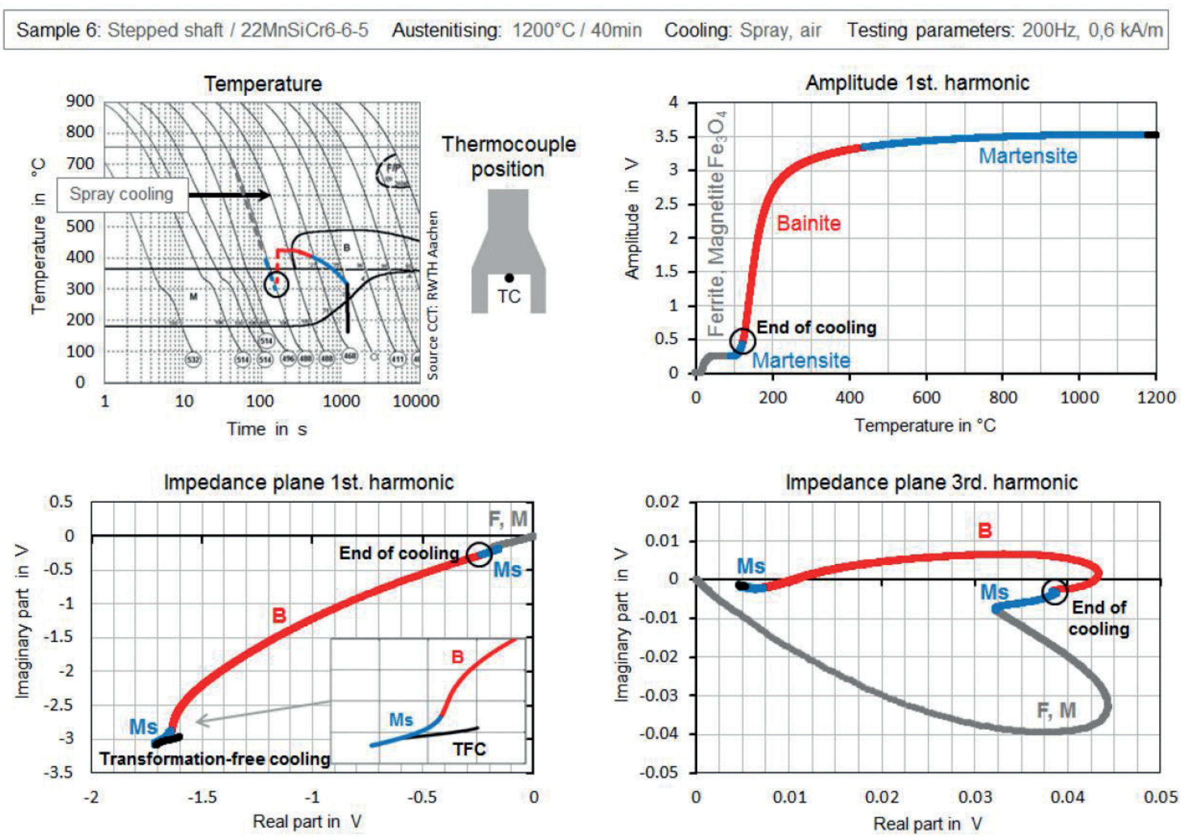

Fig. 10 - Temperature-time-path and signal evolution for sample 6 - "Stepped Shaft"

During the controlled cooling from the forging heat, the "stepped shaft" was initially cooled in the water-air spray field up to the detection of a phase transformation in the sensor's signal. On detecting the first, local initiation of martensite formation in the surface region (test frequency: 200 Hz) the spray cooling was stopped. During the subsequent cooling in static air, the residual heat from the core subsequently flows back into the surface region so that, following a slight elevation in temperature, a quasi-isothermal transformation occurs in the lower bainite region. In the 1st and 3rd harmonics, both the formation of ferrite, which is due to a decarburisation of the surface region, as well as the scaling (grey curve), can be detected during the cooling in the water-air spray field. Upon reaching the martensite start-temperature and forming of the first fractions of the martensitic microstructure (blue curve), the spray cooling was discontinued so that the martensite start-temperature had only locally been slightly exceeded for a short time. Because the nucleation had already begun, an accelerated bainite formation followed (red curve). The end of the bainitic transformation is characterised by a slight kink in the 1st harmonic in the impedance plane.

It can be seen in the magnified representation of the impedance plane that a further transformation took place after reaching the end of the transformation in the bainite region. This further transformation (blue curve) is due to the formation of some minor fractions of martensite. The end of the martensite formation is characterised by a very clear kink and a phase rotation of approx. 180° of the signal profile in the impedance plane.

ONLINE QUALITY ASSURANCE

After determining the process parameters for setting a bainitic microstructure in the demonstration component "common rail", made of 22MnSiCr6-6-5 HDB steel, a series of about 30 rail components was manufactured. The material was austenitised at a temperature of 1200 °C for 12 minutes, forged to rail components, trimmed and initially cooled using the water-air spray field.

Upon reaching the lower bainite region, the component was quickly transferred into a thermally insulated component holder in order to effect, as far as possible, an isothermal transformation in the lower bainite region. The phase transformations for the 30 components were recorded and monitored online during the process sequence for quality assurance and to investigate the precision of the forging and the repeatability of the cooling process. As examples, the signals for five rail components from the batch production are depicted in Fig. 11 (grey curves). The interruption in the signals corresponds to the duration required for transferring the component to the thermally insulated component holder.

During the batch production, it was noticed that one component exhibited a considerable deviation in its signal profile (red curve). A subsequent examination of the component's microstructure showed an approx. 1 mm wide, clearly defined inclusion of foreign material in the components core region, about 12 mm from the surface. This material had a significantly lower hardness value of approx. 200 to 250 HV1, than that of the bainitic microstructure of approx. 380 to 430 HV1. The inclusion was due to the preparation of the forging blank, during which central shrinkage cavities at the end faces of the cast block were welded using a structural-steel electrode prior to rolling. The welded parts had already been designated as defective but had erroneously not been rejected.

This example clearly demonstrates the sensitivity of the monitoring system for detecting material inhomogeneities, even at considerable depths in the component.

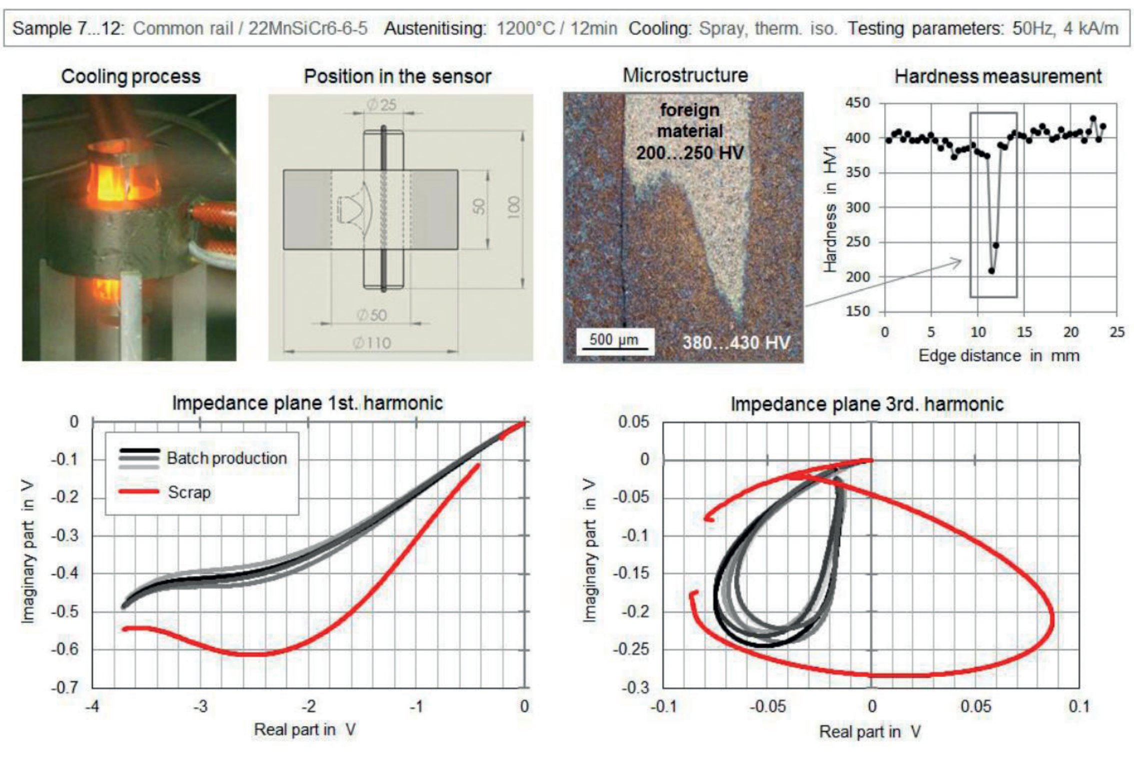

Fig. 11 - Demonstration component "common rail" and signals recorded for samples 7 to 12, see main text for details , partly recompiled from [7]

CONCLUSIONS

With the development of the new monitoring system and the corresponding high-temperature resistant sensors, new possibilities have been created for controlling, monitoring and assuring the quality of heat-treated components. Using this system, the phase transformation can be detected in situ under industrial conditions for geometrically complex components throughout their cooling phase. It was possible to show that the start, the end as well as actual microstructural state can be non-destructively and contact-free detected. Moreover, the evolution of the microstructural fractions of ferrite-pearlite, bainite and martensite can be differentiated by characteristic signal profiles, and their respective fractions can be quantified even in mixed structures. The application of the system for quality assurance was also demonstrated, and inclusions of foreign material could be clearly detected at depths larger than 10 mm.

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