Experimental determination of continuous cooling transformation diagram for high strength steel OCHN3MFA

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Abstract. Paper deals with phase transformations and austenitizing behaviour of the OCHN3MFA high strength steel. Dilatation analysis of a series of samples was performed at various cooling rates, selected in the range from 100 °C s⁻¹ to 0.01 °C s⁻¹. Acquired experimental data were used to evaluate dilatometric curves in order to map the temperature ranges of austenite to ferrite, bainite or martensite. Then, all experimental samples from dilatometric analysis, were subjected to microstructural analysis and hardness measurements to characterize the microstructure and hardness for each tested heat treatment regime.

1. Introduction
Dilatometric and plastometric analysis by using modern dilatometers is currently a widespread method for evaluation of microstructural changes and properties of metallic materials. In the case of dilatometric analysis of steels, the research focuses mainly on the study of phase transformations such as austenitization during heating, but also the conversion of austenite during cooling. The authors prefer to choose simple structure steels such as low carbon steels, micro-alloy steels or non-polymorphic austenitic and ferritic steels. OCHN3MFA steel is a medium alloyed steel based on NiCrMoV, which is characterized by a specific combination of mechanical properties – high strength and optimal level of plastic properties (toughness). The steel contains carbide-forming alloys that significantly affect phase transformations during quenching and tempering. It is intended especially for use in special technology, e.g. for the production of large-calibre barrels of howitzers or tanks.

By aniso-thermal decay of austenite we mean a process in which homogeneous austenite decays under continuous cooling. The CCT (Continuous Cooling Transform) diagrams are a graphical representation of the austenite anisothermic decay processes for a steel of a particular chemical composition. Increasing cooling rates have an analogous effect on the austenite decomposition process as well as a decreasing austenite isothermal decomposition temperature, as the diffusion conditions (temperature reduction and time to diffusion processes) deteriorate [1].

The dependence of the composition of the resulting microstructure on the cooling rate depends on the chemical composition of the steel and has been the subject of various investigations. For example, Lin et al [2] presented a paper on the effect of cooling rate on steel microstructure 30MSV6.

Dilatometers are instruments that measure an expansion curve as a function of temperature. The absolute dilatometer registers all length dimensions that occur in the sample during temperature changes. For dilatometric measurements, a change in the dimensions (volume or length) of the substance in dependence on temperature - thermal expansion is used. The nature of the thermal expansion of crystalline substances is related to the action between the atomic forces in the crystal lattice [3].
At the temperature $T_0 = 0 \, \text{K}$ the atom "2" has the minimum potential energy, the position of the atom is given by the value $a_0$. At a temperature $T_1 \neq 0 \, \text{K}$, the atom oscillates between positions $r_1$ and $r_1'$ around the mean value $a_{01}$. If we heat the body to $T_2 > T_1$, we increase the potential energy value, the atom oscillates between the positions $r_2$ and $r_2'$ around the equilibrium position $a_{02}$. Since the potential curve is asymmetric, $a_{02} > a_{01} > a_0$. If we consider the centre of the atomic path of the atom to be decisive for its position in the lattice, then we can say that with increasing temperature the interatomic distance, the lattice parameter and thus the crystal volume 

\[ \text{increase} \] [3].

The volume changes in phase transformation are due to the difference between the lattice parameter of the original and the newly formed phase. In the case of steels, this is primarily the transformation of the $\alpha$ (ferrite, K8) to the $\gamma$ phase (austenite, K12) under heating associated with austenitization and subsequently the transformation of austenite $\gamma$ to martensite, bainite or perlite upon cooling. The lattice parameter $\gamma$-iron is approximately $3.65 \cdot 10^{-10} \, \text{m}$. The value of the grid parameter – iron depends on the temperature and increases up to the value of $2.9 \cdot 10^{-10} \, \text{m}$. It is also possible to capture volume changes in the dilatometric curve in the case of phase transformations such as tetragonal martensite decay to tempering, precipitation, carbide deposition and etc. [4]. The dependence of the change in the length of the experimental sample and the temperature is called the expansion curve. In the case of a phase change, the length does not change proportionally with the temperature change, but changes due to volume changes during the conversion. From the dilatation curve, it is then possible to read the values of the temperature range in which the phase transformation takes place. In the case of steels, these are mainly the limit temperatures in the CCT diagrams, which are important in the design and optimization of heat treatment processes [4].

Dilatometer DIL805A / D is currently one of the top measuring instruments. The dilatometer 805A is used for non-deforming purposes, more specifically for heating and cooling. The temperature range is from room temperature to $1500 \, ^\circ\text{C}$ and in special cases it is possible to measure at minus temperatures, more precisely in the range from $-150 \, ^\circ\text{C}$ to $1300 \, ^\circ\text{C}$. Heating of each sample is inductive, so each sample must be electrically conductive [5]. The heating can take place in air, in a vacuum or in an internal atmosphere. Heating is possible at rate up to $4000 \, \text{K s}^{-1}$ and cooling up to $2500 \, \text{K s}^{-1}$ [6].
In practical situations, Continuous Cooling Transform (CCT) diagram play an important role in the development of high-strength advanced steels. CCT diagrams allow accurate predictions of the microstructures compositions that may arise in the real processing of these steels. They are generally used to design and optimize special heat treatments and predict the resulting microstructures and mechanical properties [7–9]. These phase transformation curves provide precise information about microstructures resulting from non-isothermal austenite decomposition. Unfortunately, the availability of CCT diagrams created for transformation of intercritical austenite practically does not exist in open literature, and therefore the mechanisms of transformation are not fully understood.

Therefore, main goal of this paper is to map phase transformation in OCHN3MFA steels with using DIL 805 dilatometer and then construct CCT diagram for this steel based on experimental results.

2. Experimental work

The basic material used in the experiments is high strength steel with the Russian designation OCHN3MFA. It is a steel with high strength and excellent plastic properties, which is used mainly for the production of main and some parts in special technology.

Chemical composition of the experimental samples was verified by spectral analyser Q4 TASMAN (table 1) and basic mechanical properties is shown in table 2.

| Table 1. Chemical composition of the OCHN3MFA examined steel (wt. %). |
|---------------------------------------------------------------|
| **Element** | **Min – Max** | **Spectral analysis** |
| C | 0.33–0.40 | 0.40 |
| Mn | 0.25–0.50 | 0.30 |
| Si | 0.17–0.37 | 0.32 |
| Cr | 1.20–1.50 | 1.19 |
| Ni | 3.00–3.50 | 3.27 |
| Mo | 0.35–0.45 | 0.52 |
| V | 0.10–0.80 | 0.13 |

| Table 2. Basic properties of the OCHN3MFA steel. |
|------------------------------------------------|
| **Mechanical and physical properties** | **Value** |
| Tensile strength $R_m$ (MPa) | 1500 |
| Modulus of elasticity $E$ (GPa) | 210 |
| Thermal conductivity (W m$^{-1}$ °C$^{-1}$) | 20 |
| Hardness (HV) | 500 |
| Specific heat (J kg$^{-1}$ °C$^{-1}$) | 460 |

2.1. Dilatometric analysis

Dilatometric analyses were performed on samples at various cooling rates from 100 °C s$^{-1}$ to 0.01 °C s$^{-1}$. The obtained experimental data were used to evaluate dilatometric curves in order to map the temperature ranges of transformation of austenite to ferrite, bainite or martensite. All experimental samples from the dilatometric analysis were subsequently subjected to the microstructural analyses and hardness measurements characteristic of each thermal treatment mode tested.

A Dilatometer DIL805A was used to investigate phase transformations. The device is equipped with a Linear Variable Differential Transformer (LVDT) sensor that measures sample length variations during the experiment [10, 11]. The experiment is carried out in a gas-tight chamber that allows testing under vacuum (heating and holding phase) or an inert atmosphere (cooling phase) to minimize oxidation.
or decarburization at high temperatures [12, 13]. The temperature is controlled and sensed by two thermocouples that are spot-welded on the sample surface.

The samples were heated to an austenitization temperature of 850 °C at a rate of 1 °C s⁻¹. Stability at austenitization temperature was 30 min. After austenitization, the samples were cooled at different rates: 0.01; 0.05; 0.1; 0.5; 1; 5; 10; 100 °C s⁻¹.

The result of the experiment was to measure eight dilatometric curves to analyse the change in sample length (ΔL) from temperature (T). A portion of the heating of the dilatometric curves was evaluated and the temperatures of Ac₁ and Ac₃ were determined. The area of cooling of the dilatometric curves was used to evaluate the decay of austenite into perlite, bainite and martensite.

Transformation temperatures were determined from individual dilatometric curves using tangential extrapolation of linear portions of the curves (ΔL / (L₀ – T)) and its corresponding first derivative (L / (L₀ / dT)). In addition to the length change curves, heating coil power curves were used, in which the coil performance is given as a percentage of temperature. The sudden change in power may be related not only to the change of magnetic properties (for example from paramagnetic austenite to ferromagnetic ferrite), but also to the transformation of individual structures in samples. The curves provided information to determine and confirm transformation temperatures.

2.2. Analysis of expansion curves
The phase change is reflected in the expansion curve as a step change in the length of the experimental sample as a function of temperature. The initial temperature Ac₁ corresponds to the temperature at which the dilatation curve begins to deviate from the linear expansion during heating due to the formation of austenite. Subsequently, the temperature Ac₃ is defined as the temperature at which the expansion curve begins to assume a linear character during heating. The heating conditions were the same for all experimental measurements. The method for determining the boundary temperatures Ac₁ and Ac₃ is shown in figure 2. Their values for the high strength steel OCHN3MFA are Ac₁ = 725 °C and Ac₃ = 763 °C.

Figure 2 shows a selected heating curve on which the beginning and end of the austenitization temperatures Ac₁ and Ac₃ are shown by tangent lines. The graph is also supplemented by a derivation curve of the given dilatation curve, which allows more accurate reading of the beginning and end of the transformation. Tangent lines overlap with the linear portion of the expansion curve, the point of beginning of the deflection of the expansion curve from tangent line indicates the beginning resp. the end of the phase transformation in the structure of the material.

The left part of the figure shows an enlarged area of the curve with the change in sample size due to phase transformation. It allows more accurate observation of the deviation of the expansion curve from the tangent lines. For comparison, we can see the derivative curve of the heating, where we can also observe the change of direction at the beginning and end of the phase change of temperatures Ac₁ and Ac₃. The derivative curve serves as one of the possibilities of determining the dilatation temperatures.

The following experimental dilatation curves present the various cooling regimes examined and describe the austenite decay and formation of the transformation phase depending on the cooling rate. This decomposition of austenite during continuous cooling is related to the temperature range of formation of a specific type of microstructure. Although the temperature ranges in which various phase transformations in the steel may occur are relatively wide and may even overlap, any phase transformation that occurs throughout the continuous cooling process can be detected from the expansion curve.

In figure 3, a dilation curve for cooling at rate of 100 °C s⁻¹ is visible. From the course of the dilatation curve and its derivative, the temperature M, was determined to 321 °C. At the stated cooling rate, only the martensitic structure and the retained austenite are present. This is also reflected in figure 11a, where the microstructure is shown after a given cooling mode.
Figure 2. Determination of transition temperatures $A_{c1}$ and $A_{c3}$ with continuous heating at 1 °C s$^{-1}$.

Figure 3. Dilatation curve for cooling mode at 100 °C s$^{-1}$.
In figure 4, we can see changes in the sample that has been cooled at 10 °C s⁻¹. Martensite formation from 294 °C was observed. The first slight decrease in temperature, which marks the start of martensite formation, is visible in the derivation curve.

The microstructure after the given cooling method (figure 11b) consists mainly of martensitic needles and retained austenite. Observation of the microstructure on an optical microscope shows that the structure contains less retained austenite than at a cooling rate of 100 °C s⁻¹.

In figures 5 and 6, the expansion curves for cooling modes of 5 °C s⁻¹ and 1 °C s⁻¹ are shown. At a cooling rate of 5 °C s⁻¹, martensite formation began from 294 °C, at a cooling rate of 1 °C s⁻¹ from 284 °C. The microstructures corresponding to these modes are shown in figures 11c and 11d. In both cases, only the martensitic transformation was recorded in the dilatation curve. By observing the microstructure in an optical microscope again, the trend of decreasing the amount of retained austenite with decreasing cooling rate was confirmed. An increase in the amount of martensite with a decrease in the cooling rate can also be deduced from the magnitude of the dimensional changes in the expansion curves that correspond to the martensitic conversion.

Then, a cooling rate of 0.5 °C s⁻¹ was evaluated (figure 7). The onset of the phase conversion of austenite to martensite is visible in the dilatation curve at 292 °C. Even after this cooling mode, the microstructure is predominantly martensitic (figure 11e). Bainitic conversion was also found in the derivative curve at sufficiently large magnification. Therefore, it can be assumed that the structure also contains a small amount of bainite.

The following cooling rate was set to 0.1 °C s⁻¹ (figure 8). At this cooling rate, the formation of two phases was detected in the expansion curve. The first phase was bainite, which began to form at 471 °C. The second phase was martensite, which began to form at 228 °C. The structure corresponding to this cooling mode consists of very fine martensitic needles and predominantly bainite (figure 11f).
**Figure 5.** Dilatation curve for cooling mode of 5 °C s⁻¹.

**Figure 6.** Dilatation curve for cooling mode of 1 °C s⁻¹.
In the penultimate dilatometric curve (figure 9) three phase transformations were detected. The first phase transformation was recorded at 657 °C, when austenite was converted to ferrite. As the temperature decreasing, another phase change occurred at 471 °C, where a bainite structure was formed. With continuous cooling of the sample, one more phase conversion to martensite occurred at a temperature of 169 °C. The structure corresponding to this cooling mode is predominantly bainite (figure 11g).
In the last cooling curve, three phase changes are again observed (figure 10). Gradually, there were three structures, ferritic, bainitic and martensitic. Ferritic structure was formed at 700 °C, bainitic at 476 °C and the last phase conversion to martensite occurred at 158 °C. At this very slow cooling rate, the microstructure consists of polygonal ferrite grains in the bainite matrix (figure 11h). Electron microscopy should be used to accurately confirm the bainite structure and its classification (upper or lower bainite).
Figure 11. The resulting microstructure after: a) 100 °C s⁻¹, b) 10 °C s⁻¹, c) 5 °C s⁻¹, d) 1 °C s⁻¹, e) 0,5 °C s⁻¹, f) 0,1 °C s⁻¹, g) 0,05 °C s⁻¹, h) 0,01 °C s⁻¹.
Cooling regimes were used to determine the average temperature $M_s$, after which only a martensitic structure (100, 10, 5, 1 and 0.5 °C s$^{-1}$) was formed. Figure 12 shows the expansion curves for these modes and compares them to determine the average temperature $M_s$. The resulting value is the average of five $M_s$ temperatures from the individual dilatation curves. The resulting value is $M = 282$ °C. The temperature $M_f$ lies in all cases in the range of negative temperatures that are outside the measuring range on DIL805A. The temperature $M_s$ slightly decreases as the cooling rate decreases.

![Diagram showing expansion curves for various cooling rates with Ms at 282°C indicated](image)

**Figure 12.** Average temperature $M_s$.

### 2.3. Determination of CCT diagram

Based on all measured dilatation curves and their analysis, the CCT diagram for OCHN3MFA steel was compiled (figure 13).

The austenitization temperature was set at 850 °C and the temperatures $A_{c1}$ and $A_{c3}$ were read from the dilatometric curve for heating. This temperature range represents the transformation of the original microstructure to austenite by heating the sample.

The diagram was created from all measured data, i.e. of all eight expansion curves. It clearly shows the areas of transformation of austenite to bainite and martensite. Subsequently, above the area of bainitic transformation, we see the region of ferrite [14–16].

The resulting temperature $M_s$ (282 °C), which is indicated by a solid black line in the diagram, was determined as the average of the temperatures $M_s$ from each cooling mode, resulting in only a martensitic structure. Since the cooling in the DIL850A chamber is limited to 50 °C, the end of the martensitic transformation $M_f$, which lies in the region of negative temperatures, was not recorded. For each cooling mode, the surface hardness of the sample was evaluated by the Vickers method with a load of $F = 49.03$ N (HV5). The resulting hardnresses are shown in figure 13 for each cooling curve. In regimes with martensitic structure the hardness decreased only slightly, but in regimes with the presence of other phases (bainite, ferrite) the hardness decrease was significantly more pronounced. The measured hardnresses correspond to the phases detected from the expansion curves as well as from the microstructure evaluation by optical microscopy.
3. Conclusion

The main objectives of this paper were dilatometric analysis of OCHN3MFA steel with focus on the influence of selected parameters on the change of structure and properties of experimental material in the monitored processes.

Dilatometric analysis of individual cooling regimes resulted in dilatometric curves (dependence of $\Delta L$ vs. $T$). Analysis of single curves revealed that only a martensitic transformation occurred at cooling rates of 100, 10, 5, 1 and 0.5 °C s$^{-1}$. Bainitic (0.1 °C s$^{-1}$) or bainitic-ferritic structure (0.05 and 0.01 °C s$^{-1}$) also occurred at slower cooling rates. These conclusions are also confirmed by microscopic analysis by optical microscopy and hardness evaluation of experimental samples after cooling. OCHN3MFA steel contains austenitic alloys (Ni, Mn) that increase the hardenability, which shift the CCT diagram lines to the right, so the martensitic structure occurs up to a cooling rate of 0.5 °C s$^{-1}$ and more equilibrium structures require very long cooling times. For example, the cooling time for the rate of 0.01 °C s$^{-1}$ was up to 20 hours. Another result of the dilatometric analysis was also the determination of limit temperatures ($A_{C1}$, $A_{C3}$, $A_{CCM}$, $M_S$), which are essentially material constants for a given chemical composition of steel. The temperature $M_f$ could not be determined in the dilatometric analysis as it lies in the negative temperature range, which was below the limit of the DIL805A dilatometer.

All data from the measured dilatometric curves and their analysis were summarized for the construction of the austenite (CCT) aniso-thermic decay of OCHN3MFA steel. The diagram was also supplemented with HV5 surface hardness values for each cooling mode. The resulting hardnesses are in accordance with theoretical knowledge. The high cooling rates characterized by the martensitic structure showed high hardness values and, on the other hand, the structures after slow cooling with the presence of bainite resp. and ferrite showed low hardness.

Figure 13. CCT diagram of OCHN3MFA steel.
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Acknowledgments
This work was supported by the Slovak Research and Development Agency under the contract No. APVV-15-0710.