Hutnické listy č. 2/2017, roč. LXX
ISSN 0018-8069

# Linear Coefficient of Thermal Expansion and Density of Steel Obtained Using Thermo-mechanical Analysis, Dilatometry and Calculations

# Lineární koeficient teplotní roztažnosti a hustota oceli stanovené pomocí termomechanické analýzy, dilatometrie a výpočtů

**Mgr. Aleš Kalup**[1,2]; **Ing. Jaroslav Beňo, Ph.D.**[3]; **Ing. Simona Zlá, Ph.D.**[1,2]; **Ing. Ľubomíra Drozdová**[1]; **Ing. Monika Kawuloková, Ph.D.**[1]; **prof. Ing. Jana Dobrovská, CSc.**[1,2]; **Ing. Ondřej Martiník**[1]; **Ing. Petr Dostál**[1]; **doc. Ing. Bedřich Smetana, Ph.D.**[1,2]

[1] VŠB – Technical University of Ostrava, Fakulty of Metallurgy and Materials Engineering, Department of Physical Chemistry and Theory of Technological Processes, 17. listopadu 15/2172, 708 33 Ostrava-Poruba, Czech Republic

[2] VŠB – Technical University of Ostrava, Fakulty of Metallurgy and Materials Engineering, Regional Materials Science and Technology Centre, 17. listopadu 15/2172, 708 33 Ostrava-Poruba, Czech Republic

[3] VŠB – Technical University of Ostrava, Faculty of Metallurgy and Materials Engineering, Department of Metallurgy and Foundry, 17. listopadu 15/2172, 708 33 Ostrava-Poruba, Czech Republic

*This paper deals with the results obtained by two experimental methods (thermo-mechanical analysis and dilatometry) and by theoretical calculations (Thermo-Calc software with TCFE8 database). Experimental data were obtained from heating runs, full chemical composition and all phases (except for graphite and diamond) were allowed for calculations. Calculations were performed in an equilibrium state.*

*Density and linear coefficient of thermal expansion of one low-carbon (0.077 wt. % C), unalloyed steel grade were investigated in wide temperature range up to 1400 °C. The results obtained by both experimental methods (TMA and dilatometry) and by theoretical calculations were compared and good agreement between the values was found. Discussion of identified differences between experimental and theoretical values was also performed.*

***Key words**: density; linear coefficient of thermal expansion; thermo-mechanical analysis; dilatometry; Thermo-Calc*

*Tato práce se zabývá stanovením hustoty a lineárního koeficientu teplotní roztažnosti nelegované nízkouhlíkové oceli (hm. %: C 0,077; Mn 0,64; Si 0,20; Cu 0,06; Cr 0,05) pomocí dvou experimentálních metod (termomechanické analýzy a dilatometrie) a teoretických výpočtů za využití programu Thermo-Calc s databází TCFE8 (Thermo-Calc Fe-based database, 8. verze) doporučenou pro výpočty vlastností ocelí.*

*Prezentované výsledky byly u obou experimentálních metod (termomechanická analýza i dilatometrie) získány pouze z ohřevu, který probíhal v inertní atmosféře argonu (čistota 6N). Výpočty podle programu Thermo-Calc byly provedeny pro rovnovážný stav. Výpočty vlastností v nerovnovážném stavu tento program neumožňuje. Do výpočtů byly z chemického složení oceli zahrnuty všechny prvky a z fází byl při výpočtech vyřazen grafit a diamant.*

*U termomechanické analýzy byla použita rychlost ohřevu vzorku 10 °C·min$^{-1}$, pro dilatometrická měření byla použita rychlost ohřevu 5 °C·min$^{-1}$. Experimentální i teoretické hodnoty relativního prodloužení i lineárního koeficientu teplotní roztažnosti byly vztaženy na stejnou referenční teplotu 25 °C.*

*Teplotní závislosti hustoty a relativního prodloužení změřené pomocí dilatometru a vypočítané s využitím programu Thermo-Calc jsou si velmi blízké. Drobné rozdíly jsou zřejmě způsobeny výpočtem v rovnovážném stavu.*

*Teplotní závislost hustoty vzorku získaná pomocí termomechanické analýzy klesá nejrychleji, což by mohlo být důsledkem nejvyšší rychlosti ohřevu (10 °C·min$^{-1}$). Teplotní závislost relativního prodloužení oceli získaná pomocí termomechanické analýzy roste nejrychleji, což je zřejmě také důsledek nejvyšší rychlosti ohřevu. Lineární koeficient teplotní roztažnosti vypočítaný pomocí programu Thermo-Calc je v celém teplotním intervalu nižší než u obou experimentálních metod, což je pravděpodobně také důsledek výpočtů v rovnovážném stavu.*

***Klíčová slova**: hustota; lineární koeficient teplotní roztažnosti; termomechanická analýza; dilatometrie; Thermo-Calc*

Thermo-mechanical analysis (TMA) and dilatometry are common experimental methods used for measurements of temperature dependence of change in the length of the sample. Change in the length of the sample is caused by changes in the lattice parameters, and this change can be described by coefficient of thermal expansion. It is generally very sensitive to composition, [1].

Density is one of the fundamental physical properties of material. Density depends not only on the chemical composition, but it also significantly depends on previous history of the steel (i.e. mechanical and/or heat processing) [2].

TMA and dilatometry records the length changes that occur during the heat treatment of a sample. Length changes can be used for determination of the coefficient of thermal expansion of the sample.

Thermal expansion is a fundamental material property, which relates dimensional changes of material to the temperature. A convenient measure of thermal expansion is the mean linear coefficient of thermal expansion and it is defined as the equation (1) [3]:

$$\alpha = \frac{L_2 - L_1}{L_0(T_2 - T_1)} = \left(\frac{\Delta L}{L_0}\right)\frac{1}{\Delta T}, \quad (1)$$

where $L_1$ (μm) and $L_2$ (μm) are the specimen lengths at the temperatures $T_1$ (°C) and $T_2$ (°C), respectively. $L_0$ (μm) is the initial specimen length and $\alpha$ ($K^{-1}$) is the linear coefficient of thermal expansion. Very often, the relationship between the length of the sample and linear coefficient of thermal expansion is given by the equation (2) [4]:

$$L_2 = L_1(1 + \alpha\Delta T). \quad (2)$$

The relative change in length as a function of temperature obtained from a dilatometer can be used to determine the phase transformation(s) during the heat treatment [5]. Knowledge of the coefficient of thermal expansion is very useful for investigation of the internal stress [6]. Another field, where coefficient of thermal expansion plays very important role, is coating of steels [7].

Besides experimental measurements of density and linear coefficient of thermal expansion, these quantities are studied also theoretically for many decades and this theme is still highly topical [8 - 10]. Theoretical calculations using specialized software (i.e. Thermo-Calc [11]) are cheap and fast, but they have some restrictions (i.e. chemical composition, phases, temperature range) [12].

In this paper, a real low carbon steel was investigated and its density, relative elongation and linear coefficient of thermal expansion were measured by two experimental methods (TMA and dilatometry) and theoretically calculated (Thermo-Calc software). The obtained experimental and theoretical values were compared and discussed.

## 1. Experimental

Real unalloyed low carbon-steel grade (wt. %: c 0.077; Mn 0.64; Si 0.20; Cu 0.06; Cr 0.05) was investigated. Samples were machined into the shape of cylinders, ground up and washed in acetone under ultrasound before measurements.

Measurements were done under inert atmosphere of pure argon (6N). All experimental values presented in this paper are from heating runs.

TMA measurements were performed using the device Setaram SETSYS $18_{TM}$ (Fig. 1) with vertical arrangement. Measurements were made in a wide temperature range up to 1400 °C, heating rate was 10 °C·$min^{-1}$, cylindrical samples had a length of approx. 11 mm and a diameter of approx. 5 mm (the sample mass was approx. 1.5 g).

Fig. 1 Setaram SETSYS $18_{TM}$ device used for TMA measurements
Obr. 1 Přístroj Setaram SETSYS $18_{TM}$ použitý pro TMA měření

Dilatometric measurements were performed using the NETZSCH Dilatometer DIL 402C/7 (Fig. 2) with horizontal arrangement in a wide temperature range up to 1350 °C. Heating rate was 5 °C·$min^{-1}$, cylindrical samples had a length of approx. 20 mm and a diameter of approx. 7 mm (the sample mass was approx. 6 g).

Fig. 2 NETZSCH Dilatometer DIL 402C/7 used for dilatometric measurements
Obr. 2 Přístroj NETZSCH Dilatometer DIL 402C/7 použitý pro dilatometrická měření

Hutnické listy č. 2/2017, roč. LXX
ISSN 0018-8069

## 2. Calculations

Calculations were performed using Thermo-Calc software with TCFE8 (Thermo-Calc Fe-based alloys) database [13]. All elements and all phases (except for graphite and diamond) were included into calculations.

Theoretical values of density were calculated according to the well-known equation (3):

$$\rho = \frac{m}{V}, \quad (3)$$

where $\rho$ (g·cm$^{-3}$) is density, $m$ (g) is weight and $V$ (cm$^3$) is volume.

Thermo-Calc software only allows calculation of volume of the sample, so the equation (4) [14] was used for calculations of relative elongation ($\Delta l/l_0$).

$$\frac{dl}{l_0} = \frac{l - l_0}{l_0} = \frac{l}{l_0} - 1 = \frac{\sqrt[3]{V}}{\sqrt[3]{V_0}} - 1 = \sqrt[3]{\frac{V}{V_0}} - 1, \quad (4)$$

where $l$ (cm) is length at the temperature $T$ (°C) and $l_0$ (cm) is length at the reference temperature $T_0$ (°C), $V$ (cm$^3$) and $V_0$ (cm$^3$) are the volumes at the temperatures $T$ and $T_0$, respectively.

The linear coefficient of thermal expansion was calculated using the equation (5) [15]:

$$\alpha = \frac{\sqrt[3]{\frac{V}{V_0}} - 1}{T - T_0}, \quad (5)$$

where $V$ (cm$^3$) is volume at the temperature $T$ (°C) and $V_0$ (cm$^3$) is volume at the temperature $T_0$ (°C). The reference temperature $T_0$ was set to 25 °C (the same temperature for both experimental methods).

In the equations (4) and (5), "cubic approximation ($V = l^3$)" was used, because of almost all phases in steels have cubic crystal lattice.

## 3. Results and discussion

First investigated quantity was density of steel and its temperature dependence. Experimental (TMA and dilatometry) and theoretical temperature dependences of densities are given at Fig. 3.

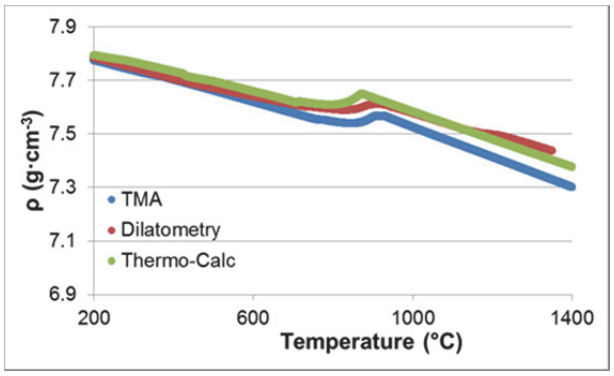


Fig. 3 Experimental (TMA and dilatometry) and theoretical (Thermo-Calc) temperature dependences of density ρ
Obr. 3 Experimentální (TMA a dilatometrie) a teoretické (Thermo-Calc) teplotní závislosti hustoty ρ

Densities obtained using dilatometry and Thermo-Calc are in excellent agreement, the difference is less than 0.05 g·cm$^{-3}$ (0.6 %). Density obtained by TMA has significantly steeper slope.

Nevertheless, the difference between TMA and both remaining methods (dilatometry and theoretical calculations) is up to 0.10 g·cm$^{-3}$ and the difference is increasing with the temperature. This might have been caused by relatively high heating rate (10 °C·min$^{-1}$) used for TMA measurements.

Small difference (up to 1.2 %) between dilatometric and theoretical values of density might have been caused by method of calculation (equilibrium state), while experiments were always non-equilibrium. Dilatometric measurements were not calibrated to temperature and this probably also affected dilatometric values.

Another investigated quantity was relative elongation of the steel sample. Experimental and theoretical relative elongations are given in Fig. 4.

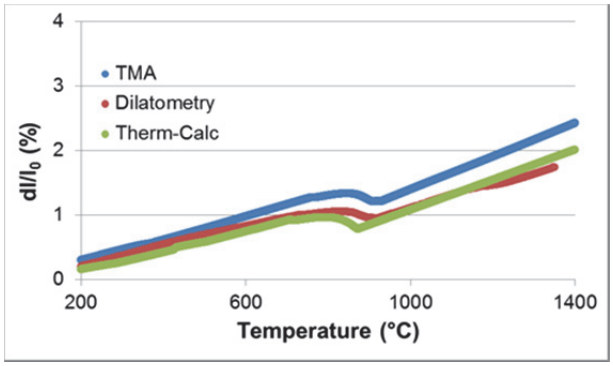


Fig. 4 Experimental (TMA and dilatometry) and theoretical (Thermo-Calc) temperature dependences of relative elongation $dl/l_0$
Obr. 4 Experimentální (TMA a dilatometrie) a teoretické (Thermo-Calc) teplotní závislosti relativního prodloužení $dl/l_0$

Relative elongation obtained by dilatometry and Thermo-Calc are in excellent agreement, the difference is no more than 0.09 %. The values obtained by TMA have faster growth, but the difference is still lower than 0.53 %. This might have been caused by different heating rate and different sample mass.

Perhaps the most valuable quantity, which was investigated, is the linear coefficient of thermal expansion. Comparison of experimental and theoretical linear coefficients of thermal expansion are given in Fig. 5.

Experimental values of linear coefficient of thermal expansion below 200 °C are influenced mainly by relatively high inaccuracy in measurements of temperature and length in this temperature area.

Theoretical values of linear coefficient of thermal expansion below 200 °C are heavily influenced by denominator in the equation (5), when T - $T_0 \rightarrow 0$. Some differences are also caused by different sample mass (approx. 1.5 g for TMA and approx. 6 g for dilatometry) and different heating rates could also have influence to measured values.

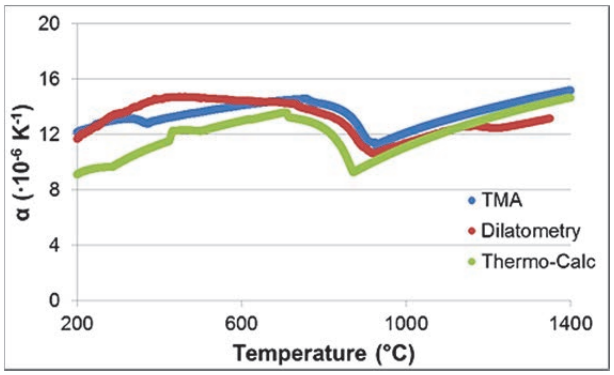


Fig. 5 Experimental (TMA and dilatometry) and theoretical (Thermo-Calc) temperature dependences of linear coefficient of thermal expansion

Obr. 5 Experimentální (TMA a dilatometrie) a teoretické (Thermo-Calc) teplotní závislosti lineárního koeficientu teplotní roztažnosti

Measured and calculated values of linear coefficient of thermal expansion above 400 °C are in good agreement, difference is up to $3.1 \cdot 10^{-6}$ $K^{-1}$. For the temperatures above 600 °C, the agreement is very good with difference less than $1.5 \cdot 10^{-6}$ $K^{-1}$.

## Conclusions

Real unalloyed low carbon steel grade was investigated and its density, relative elongation and linear coefficient of thermal expansion were obtained using two experimental methods (TMA and dilatometry) and one theoretical method (Thermo-Calc software). All three methods give similar values, except for linear coefficient of thermal expansion below 400 °C.

Values of density measured using TMA have steeper decrease than values obtained using dilatometry and calculations. Despite this, an excellent agreement between all three methods was found for density (difference is up to 0.1 $g \cdot cm^{-3}$) in the whole investigated temperature range.

Temperature dependence of relative elongation measured using TMA has steeper increase than dilatometry and calculations. Despite this, very good agreement between all three methods was found (highest difference is approx. 0.5 %) in the whole investigated temperature range.

Both experimental methods (TMA and dilatometry) give similar values of linear coefficient of thermal expansion for temperatures above 200 °C. All three used methods (TMA, dilatometry, calculations) give similar values above 600 °C, the difference is up to $1.5 \cdot 10^{-6}$ $K^{-1}$ in this temperature area.

Experimental values seem to be affected mainly by the sample mass and used heating rate. Theoretical values seem to be limited mainly by the equilibrium state calculations.

***Acknowledgement***

*This paper was prepared at the Faculty of Metallurgy and Materials Engineering within the Project No. LO1203 "Regional Materials Science and Technology Centre - Feasibility Program" funded by the Ministry of Education, Youth and Sports of the Czech Republic, GAČR project No. GA 17-18668S and student project SP2017/56.*

**Literature**

[1] DE COCK, T., CAPDEVILA, C., CABALLERO, F. G., DE ANDRÉS, C. G. Interpretation of a Dilatometric Anomaly Previous to the Ferrite-to-austenite Transformation in a Low Carbon Steel. *Scripta Materialia*, 54 (2006), 949–954.

[2] CROSS, H. C., Hill E. E. Density of Hot-rolled and Heat-Treated Carbon Steels. *Scientific Papers of the Bureau of Standards*. 22 (1927), 541–466.

[3] ELMER, J. W., OLSON, D. I., MATLOCK, D. K. Thermal Expansion Characteristics of Stainless Steel Weld Metal. *Welding Journal*, 61 (1982), 293–304.

[4] CHEN, R., MCKEEVER, S. W. S. *Theory of Thermoluminescence and Related Phenomena*. World Scientific, 1997. ISBN 978-981-02-2295-6.

[5] BROWN, M. E. *Introduction to Thermal Analysis: Techniques and Applications*. Dordrecht: Kluwer academic publishers. 2001. ISBN 978-140-20-0211-3.

[6] LI, X. Ch., STAMPFL, J., PRINZ, F. B. Mechanical and Thermal Expansion Behavior of Laser Deposited Metal Matrix Composites of Invar and TiC. *Materials Science and Engineering A*, 282 (2000), 86–90.

[7] RAMS, J., CAMPO, M., TORRES, B., UREÑA, A. Al/SiC Composite Coatings of Steel by Thermal Spraying. *Materials Letters*, 62 (2008), 2114–2117.

[8] FOILES, S. M., MURRAY, D. S. Calculation of the Thermal-expansion of Metals Using the Embedded-atom Method. *Physica Reviews B*, 38 (1988), 12643–12644.

[9] JIN, H. M., WU, P. First Principles Calculations of Thermal Expansion Coefficient: Part 1. Cubic metals, *Journal of alloys and compounds*, 343 (2002), 71–76.

[10] MATSUSHITA, T., GHASSEMALI, E., SARO, A. G., ELMQUIST, L., JARFORS, A. E. W. On Thermal Expansion and Density of CGI and SGI Cast Irons. *Metals*, 5 (2015), 1000–1019.

[11] ANDERSON, .J-O., HELANDER, T., HÖGLUND, L., SHI, P., SUNDMAN, B. Thermo-Calc & Dictra, Computational Tools for Materials Science. *Calphad*, 26 (2002), 273–312.

[12] ŽALUDOVÁ, M., SMETANA, B., ZLÁ, S., DOBROVSKÁ, J., ROSYPALOVÁ, S., KALUP, A., MICHALEK, Kl. Study of heat capacity of real steel grade. In: *Metal 2016: 25. Anniversary International Conference on Metallurgy and Materials*, 21st-23rd May 2014, Brno, Czech Republic, pp. 559–563. ISBN 978-808-72-9454-3.

[13] Thermo-Calc Software TCFE8 Steels/Fe-alloys database version 8 (accessed 23rd July 2016).

[14] KALUP, A., BEŇO, J., ZLÁ, S., KAWULOKOVÁ, M., LICHÝ, P., DOBROVSKÁ, J., DOSTÁL, P., SMETANA, B. Dilatometric Measurement and Calculation of Thermal Expansion Coefficient of Real Steel Grades. In *CEEC-TAC3 2015: 3rd Central and Eastern European Conference on Thermal Analysis and Calorimetry*, 25th-28th August 2015, Ljubljana, Slovenia, s. 205.

[15] BUJOREANU, L. G., STANCIU, S., COMĂNECI, R. I., MEYER, M., DIA, V., LOHAN, C. Factors Influencing the Reversion of Stress-induced Martensite to Austenite in a Fe-Mn-Si-Cr-Ni Shape Memory Alloy. *Journal of Materials Engineering and Performance*. 18 (2009), 500–505.