

COMPARISON OF MEASURED AND CALCULATED THERMOPHYSICAL PROPERTIES OF NICKEL SUPER-ALLOYS

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Three real grades of nickel super-alloys (IN 713LC, IN 738LC and IN 792-5A) were investigated and values of temperatures of phase transformations and latent heats of melting were obtained. All investigated quantities are very important for thermodynamic and kinetic modelling. Moreover, these data are also valuable for a lot of software used for technological processes modelling. Experimental values were obtained using Differential Thermal Analysis (DTA) measurements. Calculations were performed using Thermo-Calc 3.1 software with the use of three different databases (SSOL5, TTNi8 and TCNi6). Comparison and discussion of experimental and calculated data was performed.

Key words: nickel super-alloy, thermophysical properties, differential thermal analysis, latent heat of melting, temperatures of phase transformation

INTRODUCTION

Nickel super-alloys have many suitable properties, such as heat-resistance at high temperatures, resistance to fatigue damage, resistance to effects of flue gases, etc. A lot of data (i.e. latent heat of melting, temperature of liquidus, temperature of solidus) were measured, but there is no accessible database of thermophysical data for nickel super-alloys [1].

As for all alloys, typical necessary data for nickel super-alloys are latent heats of melting, temperatures of liquidus and solidus and other data such as specific heat. These data can be obtained using thermal analysis measurements [2] or they can be calculated by special software.

Sophisticated software, i.e. Thermo-Calc [3] includes many databases, which are necessary to its calculations. Different databases have defined different elements and they also have different maximal or minimal amount of included elements.

Databases in Thermo-Calc can be divided into two groups. “General databases”, which includes a lot of elements and restrictions for the amount of included elements are low (if any), and “specialized databases”, which includes fewer elements and restrictions for the amount of included elements are higher. Using correct database usually gives very good agreement with measured data, but still it is highly recommended to verify calculated data with available experimental data.

Thermo-Calc has two different databases designed for calculations of properties of nickel super-alloys. Comparison of results obtained using these two databases is not yet available for real nickel super-alloys.

Real nickel super-alloys contain seven or more elements [4] and the comparison was done only for systems which contain up to six elements [5].

In this paper, properties of real nickel super-alloys were calculated using three different databases (one “general database” and two “specialized databases”) and obtained results were compared to each other and also with experimental results.

MATERIAL AND EXPERIMENTAL METHODS

Three real nickel super-alloy grades [6] IN 713LC (sample 1), IN 738LC (sample 2) and IN 792-5A (sample 3) were supplied by the company PBS Velká Bíteš, a.s.

Temperatures of phase transformations and latent heats of melting were experimentally obtained from DTA measurements performed using the SETARAM Setsys 18_{TM} device.

Temperature was calibrated to the melting point of pure nickel (99,999 %) and latent heats of melting of samples were calibrated to the latent heats of melting of pure metals (the enthalpy calibration).

Temperatures of phase transformations and latent heat of phase transformations were calculated using Thermo-Calc 3.1. For nickel super-alloys, three different databases (SSOL5, TTNi8 and TCNi6) can be used to find database for which obtained values are as close as possible to the experimental values.

The SSOL5 (Solid solutions) database is the only one database which has included all elements presented in samples. The TTNi8 (Thermotech Ltd. Ni-based superalloys) and TCNi6 (Thermo-Calc Ni-based superalloys) databases are both designed for calculating the properties of nickel super-alloys, but they do not have defined sulphur and phosphorus. Both elements are very important at the γ/γ' interface [7].

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The TCNI6 database also does not have defined copper and manganese. Manganese is part of many topologically closed phases (TCP) [8], which negatively affect properties of nickel super-alloys. "Missing" elements can be added from the SSUB5 (Substances) database. In this paper, both calculations (with and without SSUB5 database extension) were performed and obtained results were compared with other experimental and calculated results.

Experimental values of temperatures of phase transformations were determined from the DTA curves. Temperatures of phase transformations were measured using various heating rates from 1 to 50 K·min⁻¹ and all obtained values were extrapolated to a zero heating rate [9]. Calculated temperatures of phase transformations were obtained from changes in the composition of phases (origination or determination of phases).

Latent heats of melting were measured with 1 K·min⁻¹ heating rate. This heating rate was chosen to be as close as possible to conditions of Thermo-Calc. Thermo-Calc only allows calculations in equilibrium, but experiments are performed in non-equilibrium.

RESULTS AND DISCUSSION

Temperature of liquidus (T_L)

Temperature of liquidus can be obtained from non-linear changes in calculated enthalpies (Figure 1). Experimentally measured (DTA) and calculated (SSOL5, TTNI8, TCNI6) T_L s are given in Table 1.

Temperatures of liquidus calculated with SSOL5 database are always higher than experimental values. For all samples, results obtained using TTNI8 and TCNI6 databases are in a very good agreement with experimental temperatures of liquidus.

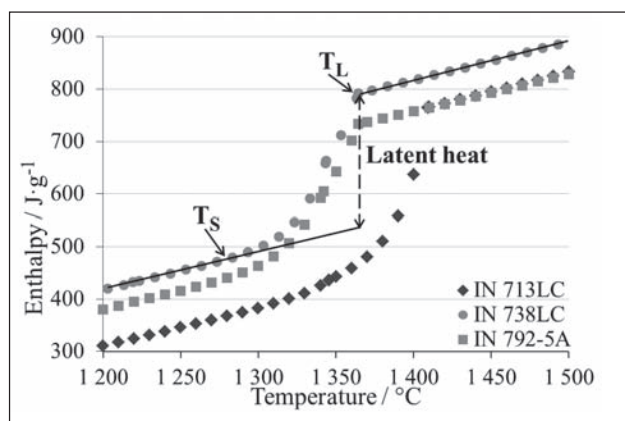


Figure 1 Calculated enthalpies using SSOL5 database

Table 1 Temperature of liquidus T_L /°C

| No. | DTA | SSOL5 | TTNI8 | TCNI6 | TCNI6* |
|-----|-------|-------|-------|-------|--------|
| 1 | 1 349 | 1 409 | 1 351 | 1 355 | 1 355 |
| 2 | 1 336 | 1 364 | 1 340 | 1 347 | 1 346 |
| 3 | 1 336 | 1 364 | 1 337 | 1 341 | 1 341 |

* with SSUB5 database extension

Values of temperature of liquidus calculated using TCNI6 database are also in a good agreement with measured temperature of liquidus and the difference between results obtained with and without SSUB5 database extension is up to 1 °C.

Temperatures of liquidus calculated with TTNI8 database are the closest to measured values for all samples. Also, T_L s calculated using TCNI6 database with SSUB5 database extension gives values a bit closer to measured values for all samples.

Temperature of solidus (T_S)

Temperature of solidus can be obtained from non-linear changes in calculated enthalpy of samples (Figure 1).

Due to the fact, that in the interval between T_S and T_L usually dissolve eutectics γ/γ' and carbides (Figure 2), T_S is often close to the temperature of dissolution of eutectics γ/γ' [5]. Also, the change in heat capacity at the T_S is gradual [10], so the value of T_S is always difficult to be determined from DTA. Because of this, temperature range is presented instead of a single value in Table 2 for DTA values.

Temperatures of solidus calculated with SSOL5 database are higher than experimental range of T_S for samples 1 and 2 and slightly above the experimental range for sample 3.

For sample 1, T_S calculated using TTNI8 database is slightly below the upper limit of experimental range. For the other two samples, values calculated using TTNI8 database are marginally above the experimental range of the temperature of solidus.

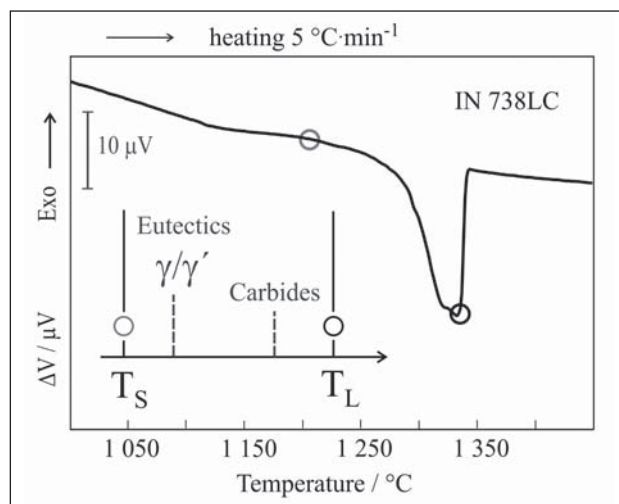


Figure 2 DTA curve of IN 738LC nickel super-alloy

Table 2 Temperature of solidus T_S / °C

| No. | DTA | SSOL5 | TTNI8 | TCNI6 | TCNI6* |
|-----|-------------|-------|-------|-------|--------|
| 1 | 1 248-1 287 | 1 346 | 1 284 | 1 270 | 1 269 |
| 2 | 1 212-1 247 | 1 294 | 1 276 | 1 243 | 1 244 |
| 3 | 1 254-1 268 | 1 270 | 1 279 | 1 245 | 1 242 |

* with SSUB5 database extension

Values obtained using TCNI6 are in the experimental range of T_s (with and without SSUB5 database extension) for samples 1 and 2. For sample 3, T_s calculated using TCNI6 database is below the experimental range (with and without SSUB5 database extension).

Latent heat of melting

Measured latent heats of melting and calculated latent heats for all samples are given in Table 3. Latent heats obtained using Thermo-Calc were calculated from its enthalpies (Figure 1).

Table 3: Latent heat of melting / J·K⁻¹

| No. | DTA | SSOL5 | TTNI8 | TCNI6 | TCNI6* |
|-----|-----|-------|-------|-------|--------|
| 1 | 193 | 289,8 | 253,9 | 286,9 | 280,5 |
| 2 | 177 | 253,1 | 261,0 | 266,3 | 266,3 |
| 3 | 163 | 236,7 | 245,2 | 246,5 | 246,4 |

* with SSUB5 database extension

Measured latent heats of melting are lower than calculated for all three studied samples. Difference between measured latent heat of melting for samples 1 and 2 is 16 J·g⁻¹ and similar difference (14,2 J·g⁻¹) is between latent heats calculated using TCNI6 database with SSUB5 database extension for this two samples. The TCNI6 database without SSUB5 database extension gives bigger difference (20,6 J·g⁻¹) between these samples.

Measured latent heat of melting for the sample 3 is 14 J·g⁻¹ below latent heat of melting of sample 2. Similar difference (16,4 J·g⁻¹) between this two samples was calculated using SSOL5 database and a little bigger difference (19,8 J·g⁻¹ and 19,9 J·g⁻¹, respectively) was calculated using TCNI6 database with and without SSUB5 database extension.

Latent heat of the first sample calculated using TTNI8 database is close to literature (264 J·g⁻¹, another computational method [11]). Latent heats calculated with other databases were higher for this sample. For sample 2, latent heat calculated using SSOL5 database is in good agreement with previously published data (248 ± 8 J·g⁻¹, DSC technique [12]).

Values of latent heat obtained with TTNI8 database are always lower than latent heats of melting calculated using TCNI6 database. Difference between latent heats of melting calculated using TCNI6 database with and without extension of SSUB5 database are up to 0,1 J·g⁻¹ for samples 2 and 3.

Measured latent heats of melting include heat effects of dissolution of eutectics γ/γ' and carbides, because both these processes occur between T_s and T_L (Figure 2). Also, many minor phases (i.e. borides [13]) dissolve in this area and their heat effects are included in the measured values of latent heats of melting.

Measured values may be also affected by many other effects – used heating rate, sample mass, aging of samples, effects that occur on the surface (i.e. oxidation). Also, DTA method is very good technique for

measuring of the phase transformation temperatures, but measurement of heat effects might be difficult to ascertain [11].

CONCLUSIONS

Temperatures of solidus and liquidus and latent heats obtained using TTNI8 and TCNI6 databases are in better agreement with experimental results than results calculated via SSOL5 database.

Latent heats, temperatures of liquidus and solidus calculated using TCNI6 database with SSUB5 database extension are same or closer to experimental results than values calculated using TCNI6 database without this extension.

Calculated latent heats were always higher than measured values. The difference between values of latent heat of samples calculated using the Thermo-Calc is similar to the difference between measured latent heat of melting of samples.

Results obtained using TTNI8 database and TCNI6 database with SSUB5 extension are almost equivalent.

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