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Kobasko, Nikolai

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Kontakt/Contact

ZBW – Leibniz-Informationszentrum Wirtschaft/Leibniz Information Centre for Economics
Düsternbrooker Weg 120
24105 Kiel (Germany)
E-Mail: [rights\[at\]zbw.eu](mailto:rights[at]zbw.eu)
<https://www.zbw.eu/econis-archiv/>

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Nikolai Kobasko

COOLING PROCESS OPTIMIZATION DURING HARDENING STEEL IN WATER POLYALKYLENE GLYCOL SOLUTIONS

Objects of investigations are water solutions of polyalkylene glycol (PAG) which are used as the quenchants in the heat-treating industry. They are tested by standard cylindrical probe made of Inconel 600 material. The main and not solved yet is the problem of transition from data achieved for standard probe to data suitable for any form and size of real steel part. It opens possibility to make predictable calculations. Taken this into account, it has been investigated water solutions of PAG of different concentration. It is underlined that cooling intensity of quenchant can be evaluated by Kondratiev number Kn . The mentioned number Kn varies within $0 \leq Kn \leq 1$ when generalized Biot Bio number varies within $0 \leq Bio \leq \infty$. As a main achievement of investigation is established correlation between standard Kn number and Kn number of real steel part. In many cases, when film boiling is absent, the established correlation is a linear function. It allows optimizing quenching processes: obtain high surface compressive residual stresses, save alloy elements and improve environment condition. All of this is achieved by tolerating chemical composition of steel with size and form of quenched object as it is proposed by UA Patent No. 114174. Also, the number Kn allows interruption of quench process when surface compressive residual stresses are at their maximum value that essentially improves the quality of steel components. Moreover, interrupted cooling prevents quench crack formation, decreases distortion of quenched steel parts. The results of investigations can be used by engineers in the heat-treating industry and scientists for further research.

Keywords: quench process, Kondratiev number Kn , cooling time, chemical composition, water solutions of polyalkylene glycol (PAG).

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1. Introduction

As discussed in references [1–3], there are several ASTM Standards for testing cooling intensity of liquid quenchants. They all are based on cooling temperature – time curves and core cooling rate curves versus core temperature of the standard cylindrical probe [3–5]. The diameter of standard cylindrical probe is 12.5 mm and its length is 60 mm which is made from Inconel 600 material and is instrumented with one thermocouple at its center [1, 2]. The designed standards allow maintaining liquid quenchants in stable condition and provide their perfect control by comparing cooling curves with each other. Such approach works perfectly for industrial oils and elevated concentration polymers in water where, as a rule, cooling process lasts almost to bath temperature. Nowadays, in heat treating industry intensive quenching (IQ) process is used that requires cooling time interruption at proper time depending on chemistry of steel and condition of cooling [6, 7]. To design computer program for cooling time interruption suitable for any steel, and any form and size of hardened product, heat transfer coefficients (HTC) are required for each specific condition of steel hardening. The task is rather complicated connected with the solving of inverse problem (IP) which belongs to

ill-posed problem [8]. When the thermocouple is located at the center of the probe, incorrect results of calculations of HTC occur. Moreover, nobody knows how obtained such a way HTC can be transferred for complicated steel parts made of different materials. There were many unsuccessful attempts to perform transition from standard probe to real steel part. The problem is not solved yet and each specific technology every time requires additional costly experiments. Author of the paper proposes a simple method of transition from standard probe to real steel parts of any form and size which could be made from different materials having different thermal properties.

As known, there are numerous of experiments related to cooling curves recordings. Many of them are published in different scientific journal and technical books [3–5]. This paper analyzes very accurate experimental data provided by ASTM Standard D6482-06 related to testing of aqueous Poly(Alkylene Glycol) solutions that form thin surface polymeric layer during quenching of heated to high temperature products. For summarizing and generalization of many published experiments, the dimensionless number Kn was used which is considered by regular thermal condition theory [9, 10]. The advantage of the theory consists in considering any form and any size of steel part

subjected to heating and cooling processes. This problem is considered below and examples of the quench process optimization, when quenching in PAG water solutions, are discussed in the paper. Thus, it is relevant to investigation is cooling intensity of polymeric quenchants which is presented by Kondratiev member Kn . It is widely used for optimizing chemical composition of steel, for cooling time interruption, and calculating cooling rate during quenching.

Therefore, *objects of investigations* are water solutions of polyalkylene glycol (PAG) which are used as the quenchants in the heat-treating industry. And *the aim of investigations* is cooling process optimization during hardening steel in water polyalkylene glycol solutions.

2. Methods of research

As known, quenching in liquid media is rather complicated process which requires further painstaking investigations. There are so many parameters affecting cooling process that in many cases makes impossible HTC generalization. However, there is a simplified method of quench process generalization based on use of dimensionless Kondratiev number Kn that presents cooling intensity of quenchant and varies within $0 \leq Kn \leq 1$ [9, 10]. When Kn is small, the cooling process is slow. When Kn is equal 1, the cooling process is ideal and cannot be more affected by increasing intensity of coolant. Taking this fact into account, the author of [7] proposed generalized equation for heating and cooling time calculation presented in the very simple form:

$$\tau = E_{eq} \frac{K}{aKn}, \quad (1)$$

where τ is cooling time in seconds; K is Kondratiev form coefficient; a is thermal diffusivity of steel in m^2/s ; E_{eq} is a value depending on generalized Biot number Bi_v and dimensionless temperature $\theta = \frac{T - T_s}{T_o - T_s}$ suitable for real Kondratiev number Kn and $\theta = \frac{T - T_m}{T_o - T_m}$ suitable for effective Kondratiev number Kn ; T_o is initial temperature; T is current temperature; T_s is saturation temperature; T_m is bath temperature. The ratio $N = \frac{T_o - T_m}{T - T_m}$ is often used for simplified cooling time evaluation.

Knowing N and Bi_v , one can evaluate E_{eq} . The proposed method of calculation is used to fix maximal surface compressive residual stresses after interruption of cooling process (Fig. 1).

Fig. 1 shows that cooling process in liquid quenchant should be interrupted when surface compressive stress reaches its maximum value and hardened layer is optimal [7].

The next problem is related to prediction of microstructure and hardness at the core of any form and size of quenched steel part based on universal equation (2) that provides cooling rate v during quenching [6, 7, 10].

$$v = \frac{aKn}{K}(T - T_m). \quad (2)$$

Calculated by equation (2) cooling rate is compared to cooling rate of Jominy standard probe that allows prediction hardness and microstructure at the core of any steel part. More detailed information is provided in the book [11].

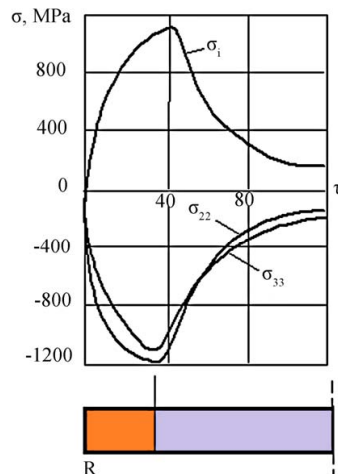


Fig. 1. Stresses at the surface of a cylindrical specimen (\varnothing 60 mm) versus time at through-hardening: orange color is martensite; gray color is intermediate phase [8, 10]

Kondratiev number Kn is also required for optimizing chemical composition of steel to provide optimal hardened layer and maximal surface compression residual stresses after quenching of products. Dimensionless number Kn is one of most important parameters that impacts a depth of hardened layer in quenched steel part [7]:

$$\frac{DI}{D_{opt}} Kn^{0.5} = 0.35 \pm 0.095, \quad (3)$$

where DI is critical diameter of cylinder that is quenched through at a given chemical composition of steel; D_{opt} is a real thickness of cylindrical part that requires formation maximal compressive surface residual stresses and optimal hardened layer; Kn is dimensionless number related to a coolant where specific cylindrical part is hardened. For complicated steel parts, equation (3) contains Kondratiev coefficients K . According to author of [12] critical diameter DI depends on chemical composition of steel and is evaluated as:

$$DI = DI_{base} \cdot f_{Mn} \cdot f_{Si} \cdot f_{Cr} \cdot f_{Ni} \cdot f_{Cu} \dots \quad (4)$$

where f_x is the multiplicative factor for the particular alloying element [12].

From considered above equations is clearly seen that the dimensionless number Kn is a base parameter which should be measured for many industrial quenchants.

3. Research results and discussion

3.1. Kondratiev Kn numbers for 5 % NaOH water solution to be compared with PAG water solutions. According to author of [13], the time of cooling from 875 to 150 °C is almost the same for different sizes and forms of steel components (Table 1) and it varies within 0.6–1 sec.

This phenomenon can be explained by an extremely fast cooling during the immersion of hot steel parts into cold liquid when film boiling is completely absent. At the very beginning of intense cooling, independently on form and size of steel part its surface is considered as a semi – infinite domain because at the beginning a very thin surface layer of a material is firstly activated. Further, when a transient nucleate boiling process is established, the cooling time essentially depends on the form and size of steel part.

Table 1 Time required for the surface of steel spheres of different sizes to cool to different temperatures when quenched from 875 °C in 5 % water solution of NaOH at 20 °C agitated with 0.914 m/s [13]

| Size, mm | Time, sec | | | | | | | |
|----------|-----------|--------|--------|--------|--------|--------|--------|--------|
| | 700 °C | 600 °C | 500 °C | 400 °C | 300 °C | 250 °C | 200 °C | 150 °C |
| 6.35 | 0.027 | 0.037 | 0.043 | 0.051 | 0.09 | 0.15 | 0.29 | 0.69 |
| 12.7 | 0.028 | 0.042 | 0.058 | 0.071 | 0.11 | 0.15 | 0.26 | 0.60 |
| 120.6 | 0.043 | 0.066 | 0.09 | 0.12 | 0.17 | 0.21 | 0.29 | 0.95 |

As seen from Table 1, quenching steel probes in 5 % NaOH water solution at 20 °C is very intensive since surface temperature of probes drops from 875 to 150 °C within one second.

To be sure that cooling process was intensive, direct and inverse problems were solved for standard cylindrical probe 12.5 diameter. For this purpose, the surface temperature of the probe was reconstructed using equation (5) that evaluates duration of transient nucleate boiling process [7, 14]:

$$\tau_{nb} = \bar{\Omega} k_F \frac{D^2}{a}, \tag{5}$$

where $\bar{\Omega}$ is a value which depends on initial temperature and characteristics of the cooling system; k_F is form coefficient; D is the thickness of quenched sample; a is thermal diffusivity of material.

Using reconstructed surface temperature from Fig. 2, inverse problem (IP) was solved to evaluate the real and effective Kondratiev numbers Kn which are presented by Fig. 3. Note, that real heat transfer coefficient (HTC) is calculated from the ratio $\alpha_{real} = q / (T - T_s)$ while effective HTC is evaluated as $\alpha = q / (T - T_m)$ [6]. Knowing HTC , generalized Biot numbers and dimensionless Kn numbers were calculated (Fig. 3). As is well known, the number Kn is the universal correlation (6) which is true for any form and size of steel part [9, 10].

$$Kn = \frac{Bi_V}{(Bi_V^2 + 1.437 Bi_V + 1)^{0.5}}, \tag{6}$$

where $Bi_V = \frac{\alpha}{\lambda} K \frac{S}{V}$; λ is thermal conductivity of steel in W/m·K; S is surface in m²; V is volume in m³.

As one can see from Fig. 3, real dimensionless number Kn during quenching in 5 % water solution of NaOH at 20 °C and agitated with 0.914 m/s is equal to 0.9 while effective Kn_{eff} is equal to 0.6. The true value for calculating intensity of cooling is Kn_{real} .

According to [6], for conventional quenching process number Kn is approximately within $0 \leq Kn \leq 0.2$ while for intensive quenching process Kn is within $0.8 \leq Kn \leq 1$. It means that 5 % NaOH water solution at 20 °C provides intensive cooling within the interval 875–100 °C where nucleate boiling takes place. These obtained results will be further compared with results of calculations related to PAG solutions.

It was shown by author of [13] that duration of transient nucleate boiling process during quenching in 5 % water solution of NaOH and 1 % water solution of PAG is the same. It means that 1 % water solution of

PAG provides intensive and uniform cooling and is environmentally green. Author of the paper analyses here 5 % water solution of NaOH, as the most uniform and intensive quenching, to compare it every time with the intensity of cooling of water PAG solutions considered below. That is very important for the practice since one can see how intense process of cooling in specific PAG water solution is as compared with 5 % NaOH.

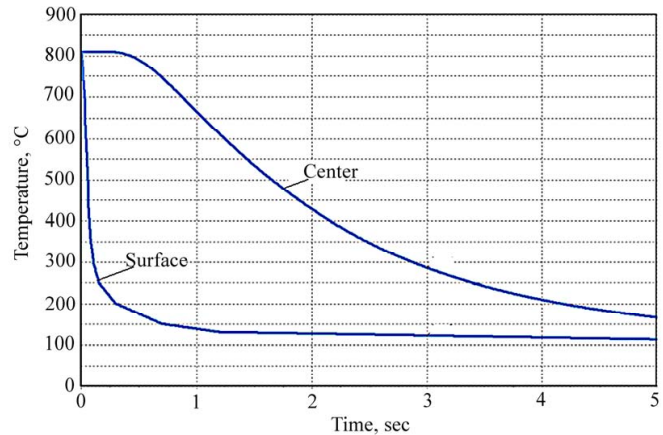


Fig. 2. Surface and core cooling curves vs time for 12.5 mm cylindrical probe during quenching from 875 °C in 5 % water solution of NaOH at 20 °C agitated with 0.914 m/s

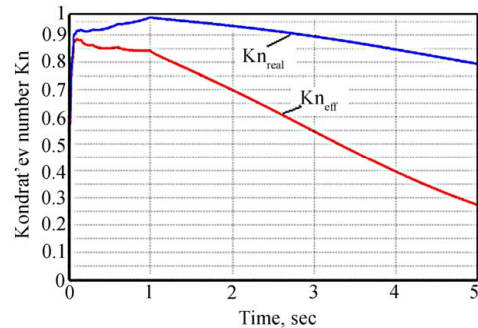


Fig. 3. Kondratiev numbers Kn_{real} and Kn_{eff} versus time during quenching of the cylindrical probe 12.5 mm diameter from 875 °C in 5 % NaOH slowly agitated water solution at 20 °C

3.2. Kondratiev Kn numbers for PAG quenchant at 30 °C and agitation 0.5 m/s depending on its concentration.

ASTM Standard D 6282-06 provides very accurate cooling temperature – time and cooling rate curves showing effect of concentration on cooling curve performance for Poly(Alkylene Glycol) quenchant at 30 °C and 0.5 m/s agitation [1]. These very important results of experiments are shown in Fig. 4.

Experimental results illustrated in Fig. 4 show no film boiling at all when quenching in 5 % and 10 % PAG water solutions. Deformation of cooling rate curves is observed when quenching in 15 % and 20 % PAG water solutions. Absence of film boiling process during quenching in PAG solutions is explained by creation of surface polymeric insulating layer with the reduced thermal conductivity (Fig. 5). As known [14], insulation results in decreasing initial heat flux density which after insulation drops below the critical heat flux density, i. e. $q_{in} < q_{cr1}$.

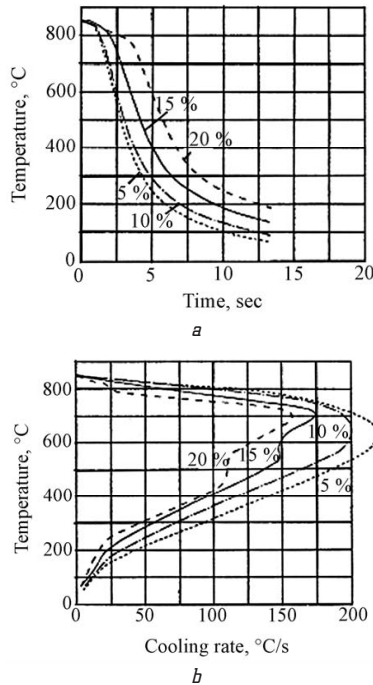


Fig. 4. Illustration of the effect of quenchant concentration on cooling curve performance for PAG solutions at 30 °C and agitation 0.5 m/s [1]: a – cooling curves versus time; b – cooling rate versus time

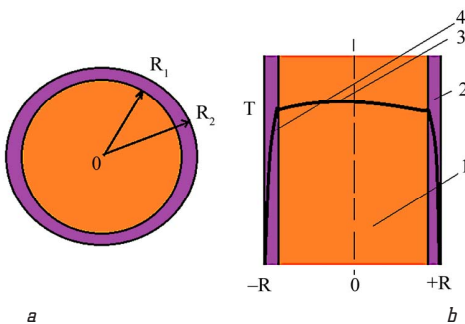


Fig. 5. Section of a coated cylindrical probe and typical temperature distribution during quenching in polymer water solution of inverse solubility: a – section of a cylinder; b – temperature distribution in cylinder; 1 – heated metal, 2 – coating, 3 – temperature gradient in metal, 4 – temperature gradient in polymeric surface layer

Formation of surface insulating layer during quenching in inverse solubility polymers was observed by accurate experiments of author of [15].

Decrease of initial heat flux density q , due to insulating layer of thickness δ , is calculated by equation [14]:

$$q_{in} = \frac{q_0}{\left(1 + 2 \frac{\delta}{R} \frac{\lambda}{\lambda_{coat}}\right)} \quad (7)$$

where q_0 initial heat flux density without insulating layer; R is radius of cylinder; λ is thermal conductivity of steel; λ_{coat} is thermal conductivity of insulating layer (coating); $\chi = 1 + 2 \frac{\delta}{R} \frac{\lambda}{\lambda_{coat}}$.

Table 2 presents value χ versus thickness of insulating layer and thickness of cylindrical probe to see how initial heat flux density is decreased with changing of insulating layer and thickness of probe.

Table 2

Value χ versus thickness of insulating layer and thickness of cylindrical probe

| D , mm | δ , mm | | | |
|----------|---------------|-------|------|------|
| | 0.001 | 0.01 | 0.05 | 0.1 |
| 10 | 1.04 | 1.4 | 3 | 5 |
| 20 | 1.02 | 1.2 | 2 | 3 |
| 50 | 1.008 | 1.08 | 1.4 | 1.8 |
| 80 | 1.005 | 1.05 | 1.25 | 1.5 |
| 100 | 1.004 | 1.04 | 1.2 | 1.4 |
| 200 | 1.002 | 1.02 | 1.1 | 1.2 |
| 300 | 1.001 | 1.013 | 1.07 | 1.13 |

As was mentioned above, the absolutely correct solution of inverse problem (IP) exploring one thermocouple at the center of cylindrical standard probe is impossible. One can evaluate only approximate values of dimensionless numbers Kn which can be used in the practice only for core cooling time and core cooling rate evaluation. These values must be considered as the empirical data suitable for cooling time interruption when core temperature relates to maximum surface compression residual stresses, and so on. The simplified method of calculation was considered in many publications and is summarized in [6, 9, 10]. Real and effective Kondratiev numbers are evaluated as:

$$Kn_{real} = \frac{vK}{a(T - T_s)}; \quad (8)$$

$$Kn_{eff} = \frac{vK}{a(T - T_m)}; \quad (9)$$

where T_s is saturation temperature; T_m is bath temperature; $K = \frac{R^2}{\pi^2}$ is Kondratiev form coefficient for standard cylindrical probe 12.5 mm diameter. It can be rewritten as $K = 10.13 \cdot 10^{-6} \text{ m}^2$.

Average HTC is calculated using equation:

$$\alpha = \frac{\lambda Bi_V V}{KS}. \quad (10)$$

Thermal conductivity and thermal diffusivity of Inconel 600 versus temperature are presented in references [6, 11].

Using well known thermal properties of Inconel 600, dimensionless numbers Kn and HTC were calculated which are presented in Tables 3–7.

Table 3

Effect of PAG concentration on maximum cooling rate v_{max} , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC when quenching standard probe in solution at 30 °C

| Concentration, % | 5 % | 10 % | 15 % | 20 % |
|----------------------------|-------|-------|-------|-------|
| V_{max} , °C/s | 218 | 200 | 174 | 158 |
| Kn | 0.598 | 0.527 | 0.414 | 0.376 |
| Bi_V | 1.25 | 0.958 | 0.63 | 0.54 |
| HTC , W/m ² K | 8930 | 8460 | 5750 | 4930 |

Table 4

Effect of PAG concentration on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC when quenching standard probe in solution at 30 °C and at probe core temperature 600 °C

| Concentration, % | 5 % | 10 % | 15 % | 20 % |
|-------------------------------|-------|-------|-------|-------|
| $V, \text{ }^\circ\text{C/s}$ | 216 | 197 | 148 | 127 |
| Kn | 0.637 | 0.581 | 0.437 | 0.375 |
| Bi_V | 1.45 | 1.17 | 0.682 | 0.54 |
| $HTC, \text{ W/m}^2\text{K}$ | 12640 | 10200 | 5960 | 4710 |

Table 5

Effect of PAG concentration on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC when quenching standard probe in solution at 30 °C and at probe core temperature 500 °C

| Concentration, % | 5 % | 10 % | 15 % | 20 % |
|-------------------------------|-------|-------|-------|-------|
| $V, \text{ }^\circ\text{C/s}$ | 187 | 163 | 137 | 110 |
| Kn | 0.713 | 0.619 | 0.521 | 0.418 |
| Bi_V | 2.00 | 1.35 | 0.710 | 0.638 |
| $HTC, \text{ W/m}^2\text{K}$ | 16560 | 11180 | 5880 | 5280 |

Table 6

Effect of PAG concentration on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC when quenching standard probe in solution at 30 °C and at probe core temperature 400 °C

| Concentration, % | 5 % | 10 % | 15 % | 20 % |
|-------------------------------|-------|-------|-------|-------|
| $V, \text{ }^\circ\text{C/s}$ | 143 | 118 | 97 | 92 |
| Kn | 0.754 | 0.622 | 0.511 | 0.485 |
| Bi_V | 2.44 | 1.37 | 0.90 | 0.82 |
| $HTC, \text{ W/m}^2\text{K}$ | 19110 | 10730 | 7050 | 6420 |

Table 7

Effect of PAG concentration on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC when quenching standard probe in solution at 30 °C and at probe core temperature 300 °C

| Concentration, % | 5 % | 10 % | 15 % | 20 % |
|-------------------------------|-------|-------|-------|-------|
| $V, \text{ }^\circ\text{C/s}$ | 93.5 | 73 | 56 | 40 |
| Kn | 0.77 | 0.602 | 0.461 | 0.404 |
| Bi_V | 2.65 | 1.25 | 0.74 | 0.60 |
| $HTC, \text{ W/m}^2\text{K}$ | 19615 | 9250 | 5480 | 4440 |

Fig. 6 shows dependence dimensionless number Kn from PAG concentration when temperature at the center of standard probe is 600 °C. According to Fig. 6, Kn number decreases from 0.64 to 0.38 when concentration of PAG increases from 5 to 20 %.

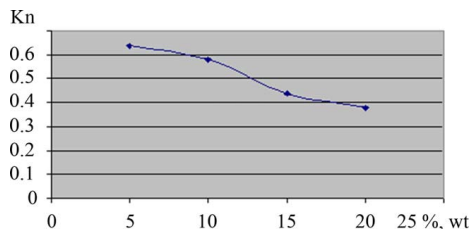


Fig. 6. Dimensionless number Kn versus PAG concentration when temperature at the center of standard probe is 600 °C

Investigations showed that dimensionless number Kn with decreasing probe core temperature changes insignificantly.

Insignificant increase Kn with decreasing core temperature of standard cylindrical probe is explained by dissolving the surface insulating later that increases heat flux and consequently increases HTC and dimensionless numbers Bi_V and Kn .

3.3. Effect of bath temperature on dimensionless numbers Kn for 15 % aqueous solution Poly(Alkylene Glycol) Quenchant.

As known, increase temperature of a quenchant decreases critical heat flux densities resulting in film boiling formation. It is important for the practice to see this effect when exploring inverse solubility polymers. Temperature – time and cooling rate curves for 15 % PAG solution are presented in Fig. 7 [1].

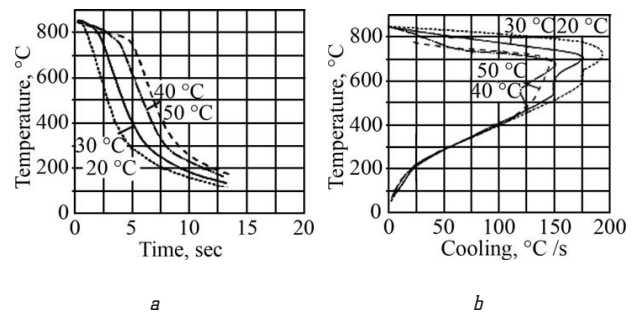


Fig. 7. Illustration of the effect of bath temperature on cooling curve performance for 15 % aqueous PAG solution at 0.5 m/s [1]: a – cooling curves versus time; b – cooling rate versus time

Effect of bath temperature on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC for 15 % aqueous solution Poly(Alkylene Glycol) Quenchant at 0.5 m/s agitation and at probe core temperature 600 °C is shown in Table 8.

Table 8

Effect of bath temperature on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC for 15 % aqueous PAG quenchant at 0.5 m/s agitation and at probe core temperature 600 °C

| Temperature, °C | 20 °C | 30 °C | 40 °C | 50 °C |
|-------------------------------|-------|-------|-------|-------|
| $V, \text{ }^\circ\text{C/s}$ | 172 | 148 | 129 | 138 |
| Kn | 0.516 | 0.444 | 0.387 | 0.414 |
| Bi_V | 0.918 | 0.70 | 0.562 | 0.623 |
| $HTC, \text{ W/m}^2\text{K}$ | 8070 | 6150 | 4940 | 5476 |

Fig. 8 shows effect of bath temperature on Kondratiev number Kn for 15 % aqueous solution Poly(Alkylene Glycol) Quenchant related to core temperature of standard probe 600 °C when agitation is 0.5 m/s.

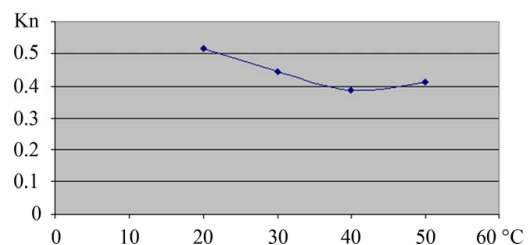


Fig. 8. Effect of bath temperature on Kondratiev number Kn for 15 % aqueous PAG solution related to core temperature of standard probe 600 °C (agitation 0.5 m/s)

Similar results of calculations obtained for core temperature 500 °C (Tables 9–11). However, dimensionless number doesn't change for core temperature 300 °C (Fig. 9).

Table 9

Effect of bath temperature on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC for 15 % aqueous PAG solution at 0.5 m/s and at probe core temperature 500 °C

| Temperature, °C | 20 °C | 30 °C | 40 °C | 50 °C |
|----------------------------|-------|-------|-------|-------|
| V , °C/s | 143 | 136 | 122 | 129 |
| Kn | 0.474 | 0.449 | 0.403 | 0.426 |
| Bi_V | 0.79 | 0.71 | 0.60 | 0.65 |
| HTC , W/m ² K | 7930 | 7130 | 6020 | 6525 |

Table 10

Effect of bath temperature on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC for 15 % aqueous PAG solution at agitation 0.5 m/s and at probe core temperature 400 °C

| Temperature, °C | 20 °C | 30 °C | 40 °C | 50 °C |
|----------------------------|-------|-------|-------|-------|
| V , °C/s | 102 | 100 | 98 | 97 |
| Kn | 0.51 | 0.50 | 0.49 | 0.485 |
| Bi_V | 0.90 | 0.88 | 0.84 | 0.82 |
| HTC , W/m ² K | 8200 | 8020 | 7656 | 7470 |

Table 11

Effect of bath temperature on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC for 15 % aqueous PAG solution at agitation 0.5 m/s and at probe core temperature 300 °C

| Temperature, °C | 20 °C | 30 °C | 40 °C | 50 °C |
|----------------------------|-------|-------|-------|-------|
| V , °C/s | 56 | 56 | 56 | 56 |
| Kn | 0.42 | 0.42 | 0.42 | 0.42 |
| Bi_V | 0.64 | 0.64 | 0.64 | 0.64 |
| HTC , W/m ² K | 5270 | 5270 | 5270 | 5270 |

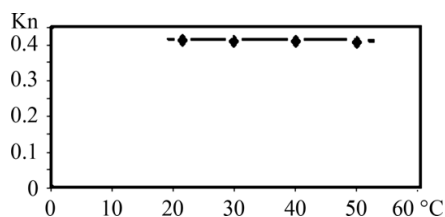


Fig. 9. Effect of bath temperature on Kondratiev number Kn for 15 % aqueous PAG solution at agitation 0.5 m/s related to core temperature of standard probe 300 °C

It can be explained by transition surface temperature from boiling process to convection.

3.4. Effect of agitation on cooling characteristics of 15 % aqueous PAG solutions at 30 °C. Accurate experiments provided in ASTM Standard D 6482-06 concerning cooling curves obtained during quenching standard probe in 15 % aqueous solution of PAG at 30 °C don't shown essential effect of agitation on cooling curves performance. In contrary, increase of PAG aqueous solution agitation leads to deformation of cooling rate curves (Fig. 10). To see this effect, see Tables 12, 13 which show behavior of Kn , Bi_V and HTC vs core temperature of standard probe.

Fig. 10 shows that developed full film boiling is absent and there is no essential change of cooling rate curves

vs intensity of agitation. From the point of view of thermal science, the cooling process can be considered as a cooling of a multilayer system (cylindrical probe covered by polymeric layer). The transient nucleate boiling process in this case takes place on the created polymeric layer instead of metal surface. Temperature on the polymeric surface maintains at the level of boiling point of water creating increased temperature gradient inside the polymeric layer. According to author of [16], agitation doesn't affect visibly HTC during nucleate boiling process (Tables 12–15). Agitation initiated by bubbles prevails significantly man – made agitation (frequency of vapor bubbles release is more than 60 s⁻¹). During nucleate boiling thousands of bubbles are intensively acting creating huge HTC . In such condition only change of thickness of insulating layer can affect cooling curve performance (Fig. 10).

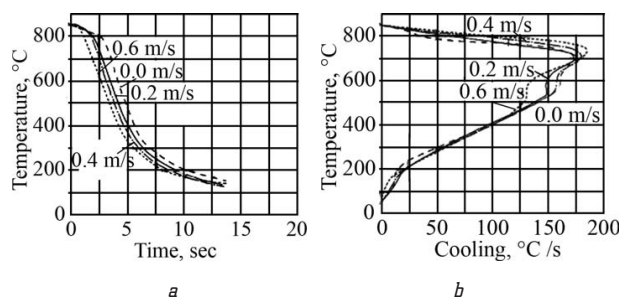


Fig. 10. Illustration of Agitation Rate on Cooling Curve Performance for 15 % Aqueous Poly(Alkylene Glycol) Quenchant Solution at 30 °C [1]: a – cooling curves versus time; b – cooling rate versus time

Table 12

Effect of agitation on maximum cooling rate v_{max} , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC for 15 % aqueous PAG solution at 30 °C

| Agitation, m/s | 0 | 0.2 | 0.4 | 0.6 |
|----------------------------|-------|-------|-------|-------|
| V , °C/s | 172 | 175 | 180 | 185 |
| Kn | 0.409 | 0.417 | 0.428 | 0.401 |
| Bi_V | 0.61 | 0.635 | 0.66 | 0.595 |
| HTC , W/m ² K | 5570 | 5795 | 6025 | 5430 |

Table 13

Effect of agitation on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC for 15 % aqueous PAG solution at 30 °C and core temperature 600 °C

| Agitation, m/s | 0 | 0.2 | 0.4 | 0.6 |
|----------------------------|-------|-------|-------|-------|
| V , °C/s | 160 | 150 | 158 | 108 |
| Kn | 0.472 | 0.443 | 0.466 | 0.319 |
| Bi_V | 0.78 | 0.877 | 0.762 | 0.43 |
| HTC , W/m ² K | 6800 | 5900 | 6645 | 3750 |

Table 14

Effect of agitation on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC for 15 % aqueous PAG solution at 30 °C and core temperature 500 °C

| Agitation, m/s | 0 | 0.2 | 0.4 | 0.6 |
|----------------------------|-------|-------|-------|-------|
| V , °C/s | 137 | 138 | 188 | 128 |
| Kn | 0.52 | 0.521 | 0.524 | 0.486 |
| Bi_V | 0.922 | 0.93 | 0.94 | 0.82 |
| HTC , W/m ² K | 7620 | 7620 | 7770 | 6780 |

Table 15

Effect of agitation on cooling rate v , Kondratiev numbers Kn , generalized Biot number Bi_V and HTC for 15 % aqueous PAG solution at 30 °C and core temperature 300 °C

| Agitation, m/s | 0 | 0.2 | 0.4 | 0.6 |
|-----------------------------|-------|-------|-------|-------|
| $V, ^\circ\text{C/s}$ | 50 | 56 | 58 | 60 |
| Kn | 0.412 | 0.461 | 0.478 | 0.494 |
| Bi_V | 0.622 | 0.756 | 0.800 | 0.800 |
| $HTC, \text{W/m}^2\text{K}$ | 4600 | 5600 | 5920 | 6220 |

As seen from Fig. 11, dimensionless number Kn at first slightly increases when agitation increases from 0 to 0.4 m/s and then abruptly decreases to $Kn=0.4$ when agitation is 0.6 m/s. It can be explained by local film boiling formation on the places where insulating layer was destroyed. This local film boiling deforms slightly cooling rate curve shown in Fig. 10 where agitation is 0.6 m/s. As known, any film boiling process is unacceptable because it results in increase distortion of quenched products. Based on this fact, it makes sense to provide agitation using low agitated hydrodynamic emitters that effectively destroy any film boiling process via resonance effect or special fixtures [14, 17].

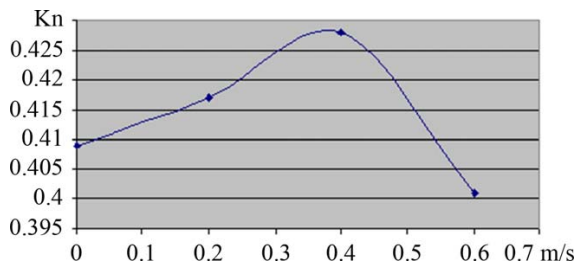


Fig. 11. Effect of agitation on Kondratiev number Kn for 15 % aqueous PAG solution at 30 °C

Thus, the results of investigations showed that 1 % aqueous PAG solutions combined with hydrodynamic emitters can be perfect tool for performing less costly intensive quenching (IQ) processes.

3.5. Evaluation Kn numbers for PAG-35 solutions. Authors of [18] investigated cooling intensity of PAG solutions with molecular mass equal to $Mm=35 \cdot 10^3$ (PAG-350). Instead of standard cylindrical probe 12.5 mm they used cylindrical probe 10 mm diameter. Different concentrations were investigated: 1, 2, 3, 5, 7 and 10 %. Some results of their investigations are presented in Table 16.

Cooling parameters obtained by testing cylindrical probe 10 mm diameter at bath temperature 20 °C with no agitation [18]

| Cooling condition | Unit | PAG-35, % wt | | | | | |
|--|------------------------------------|--------------|-----|-----|------|-------|------|
| | | 1.0 | 2.0 | 3.0 | 5.0 | 7.0 | 10 |
| Probe cooling time from 810 to 600 °C | τ_{600}, s | 1.5 | 1.4 | 1.5 | 1.6 | 2 | 1.9 |
| Probe cooling time from 810 to 400 °C | τ_{400}, s | 2.3 | 2.3 | 2.4 | 2.7 | 3.3 | 2.4 |
| Probe cooling time from 810 to 200 °C | τ_{200}, s | 3.9 | 4.0 | 4.5 | 5.6 | 7.1 | 8.0 |
| Maximum cooling rate | $V_{\text{max}}, ^\circ\text{C/s}$ | 294.5 | 252 | 251 | 220 | 202.6 | 178 |
| Core temperature at maximum cooling rate | $T, ^\circ\text{C}$ | 540 | 510 | 591 | 586 | 663 | 676 |
| Cooling rate at temperature 300 °C | V_{300} | 133.5 | 127 | 106 | 73.8 | 57.6 | 54.1 |

Using maximal cooling rates and cooling rates at 300 °C for each concentration, a correlation between Kn and PAG-35 concentration was established which is shown in Figs. 12, 13.

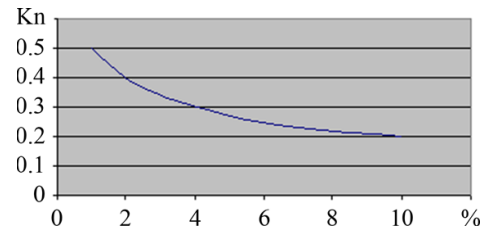


Fig. 12. Correlation between Kn and PAG-35 concentration for test Inconel 600 probe 10 mm diameter

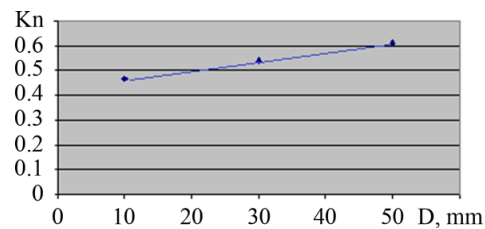


Fig. 13. Correlation between Kn and thickness of cylindrical probe when quenching in 1 % aqueous of PAG-35

For designing correlation between Kn and thickness of cylindrical probe, when quenching in 1 % aqueous of PAG-35, early published data, concerning cylindrical probe 50 mm in diameter, were used [19].

It should be noted here that form of the probe doesn't affect significantly linear correlation Kn versus time.

Thus, the simple methodic for transition from standard probe to real steel parts is elaborated in this paper. The obtained results can be used for interrupting cooling process to provide maximal surface residual stresses and fine bainitic microstructure at the core of quenched steel parts.

3.6. Discussion. Investigators, who were dealing with probes instrumented with one thermocouple at their center, lost many extremely interesting and very important for the practice phenomena. Some physical phenomena can be recorded only by the surface thermocouple. Author of paper [13] was the first who paid special attention to instrumentation of the surface thermocouples. He made very important for the practice discoveries. It was established by him that film boiling is completely absent when quenching steel samples from 875 °C in cold electrolytes.

Table 16

It was also found by mentioned author that cooling time from 875 to 150 °C is the same independently on size of the probe. These two facts made possibility for generalization experimental data and performing accurate cooling time calculation of different steel parts and made a basis for the formulation mathematical models for the quench process. It should be noted here that even leading thermal scientists have a doubt that film boiling is completely absent during quenching from 875 °C in cold liquids. It cannot be was the

answer. The big temperature difference must generate the film boiling process. Further investigators switched from surface thermocouples to central thermocouples claiming that surface thermocouple often show wrong data. The matter was that nobody could explain some very strange behavior of surface thermocouples during quenching in cold liquid media. One of them was so called the self – regulated thermal process where surface temperature during quenching maintains relatively a long time at the level of a boiling point of a liquid. Even author of [13] published his experimental data related only to interval of temperature 875–150 °C. Further the self – regulated thermal process took place which, probably, was not understandable to investigator.

Author of paper [20] did a lot of work to go back to surface thermocouples and introduce his Liščić' probe, instrumented with the surface thermocouples, to heat treating industry and scientific labs. Using Liščić' probe, some very strange behavior of surface thermocouple was observed. The surface temperature for a long time maintains at the level of 450 °C oscillating at this level while at the same time the central thermocouple was not affected by temperature delay at all [20].

Similar strange experimental results were achieved in 2019 in Germany. Authors of [21] fulfilled very accurate experiments with a shaft 150 mm in thickness and 500 mm long weighting 78 kg. Three surface thermocouples (2–4) were instrumented on the surface of the shaft and one thermocouple (1) was instrumented at the bottom of the shaft. All three thermocouples showed very stable results of measurements where surface temperature maintains for 200 seconds at the level of 300 °C.

Author of the current paper recorded similar behavior of the surface thermocouple during quenching cylindrical probe 20 mm diameters in water solutions of polymers. A simple explanation was provided regarding formation a «shoulder» during quenching steel probes in polymers.

Discovered effect on «shoulder» formation at the elevated temperatures has a great future since it allows to develop new technologies to perform low temperature thermo – mechanical treatment and perform austempering and martempering processes via cold liquids [17]. It is not costly and can be introduced to forging shops and heat-treating industry. More information on performing austempering process to obtain fine bainitic microstructure process is provided in [22, 23]. Contemporary methods of quench process optimizing are widely discussed by leading experts, e. g. in [24–26].

It should be noted here that obtained heat transfer coefficients and Kondratiev numbers Kn can be used only for approximate core cooling rate and core cooling time calculation since tests of polymer quenchants were made using standard probe with one thermocouple at its centre. To investigate accurately temperature fields during quenching and get more useful information on transient nucleate boiling processes, Liščić' probe for tests of liquid quenchants should be used which is instrumented with three tiny thermocouples [20]. Such accurate experimental data can be used then for solving inverse problem to design DATABASE for liquid quenchants [27–29]. Also, as a contemporary tool, it is recommended to use computer fluid dynamics (CFD) simulation for quench process investigation [30].

4. Conclusions

The present work proposes a generalized solution to quenching data from steel of any form and size, enabling the quench process optimization for industrial products. The work established a correlation, generally a linear function, between dimensionless number Kn of standard Inconel 600 probe and dimensionless number Kn of a real steel part. The determination of dimensionless numbers Kn enables optimization of cooling process to obtain maximum surface compression residual stresses and optimization of chemical composition of steel, depending on size and form of hardened products. Water solutions of inverse solubility polymers are unique quenchants exhibiting the possibility to create «a shoulder» acceding martensite start temperature M_s that can be used for performing austempering and martempering processes via cold liquids. Moreover, water solution (1 %) of inverse solubility PAG polymer provides uniform and accelerated cooling with the intensity $0.4 < Kn < 0.6$ for thickness of probes 10–50 mm. These facts open the great possibility to perform low temperature thermomechanical treatment in forging shops just using cold liquids. Obtained Kn numbers will be widely used to interrupt quenching at proper time to create by heat treatment process super strengthened materials.

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Nikolai Kobasko, PhD, Fellow of ASM International, Consultant, Intensive Technologies Ltd, Kyiv, Ukraine, e-mail: nkobasko@gmail.com, ORCID: <https://orcid.org/0000-0002-7636-5298>