# AN EFFECT OF PIB ADDITIVES TO MINERAL OIL RESULTING IN ELIMINATION OF FILM BOILING DURING STEEL PARTS QUENCHING

***Nikolai Kobasko***
*Intensive Technologies Ltd*
*68/1 Peremohy ave., Kyiv, Ukraine, 03113*
*nkobasko@gmail.com*

***Anatolii Moskalenko***
*Thermo- Acoustical Diagnostic of Heat Transfer Processes*
*Institute of Engineering Thermophysics of NASU*
*2A Zhelyabova str., Kyiv, Ukraine, 03057*
*an.moskalenko@gmail.com*

***Petro Lohvynenko***
*Department of Polymers Modification*
*Institute of Macromolecular Chemistry of NASU*
*48 Kharkivske road, Kyiv, Ukraine, 02160*
*petmol@ukr.net*

***Larisa Karsim***
*Department of Polymers Modification*
*Institute of Macromolecular Chemistry of NASU*
*48 Kharkivske road, Kyiv, Ukraine, 02160*
*karsim4ik@gmail.com*

***Sergii Riabov***
*Department of Polymers Modification*
*Institute of Macromolecular Chemistry of NASU*
*48 Kharkivske road, Kyiv, Ukraine, 02160*
*Sergii.Riabov@gmail.com*

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**Abstract**

To control the process of film boiling during quenching in oils, quench oil makers as a rule manipulate physical properties such as a surface tension and viscosity. However, there is much experimental data showing that special additives can eliminate film boiling in oils without changing their physical properties and which is counterintuitive. Authors explain such phenomenon by showing that the addition of a special additive, for example PIB (polyisobutylene polymer), will create an insulating layer on the surface of steel parts during quenching in oils that will eliminate film boiling without affecting physical properties of the oil. Insulating layer decreases initial heat flux density which becomes less than critical one and of the oil will not begin film boiling during quenching with the PIB additive. Authors believe that such approach will allow engineers to solve effectively the problem of part distortion after quenching. The new oil quenchant containing special additive PIB is patented in Ukraine and is manufactured by Barkor Ltd for needs of the heat treating industry.

**Keywords:** quenching, heat treating, insulating layer, film boiling elimination, distortion, no film boiling, smooth cooling benefits.

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## 1. Introduction

In the paper a patented technology is discussed [1]. In 1987 authors proposed an original idea [2] on the possibility of eliminating film boiling during quenching by creating an insulating layer on the surface of steel parts to be hardened. The invented process was rather costly that is why the author [3, 4] proposed to use small concentration of inverse solubility polymers in water which create micro layer on the surface during the beginning of the quench cooling process. In this case technology becomes significantly cheaper resulting in decreased distortion of the steel parts after quenching. Recently, authors of an original investigation [5] reported on the possibility to eliminate film boiling completely during quenching in mineral oil I-20A if PIB-2400 in amount of 3 % is added to mineral oil. Authors were sure that such phenomenon is connected with the physical properties change of oil I-20A, specifically a change to the surface tension due to presence of PIB-2400 in oil in the amount of 3 %. However, after investigating surface tension of oils with and without additives of PIB-2400, it turned out that its concentration up to 3 % does not have a significant effect on the oil's surface tension. If it is not a change to the oil's surface tension, what is a reason for eliminating film boiling in oil during quenching (see curve ACD in **Fig. 1, 2**) with the addition of the PIB-2400 additive. After discussion of the problem, authors decided to find out whether additive could create an insulating layer on the surface of Inconel 600 probe which could be a reason for such unusual behavior. Thus, the aim of this paper is to find out a reason for elimination of film boiling when PIB-2400 in oil is present by exploring the previous studies of the authors [2–4].

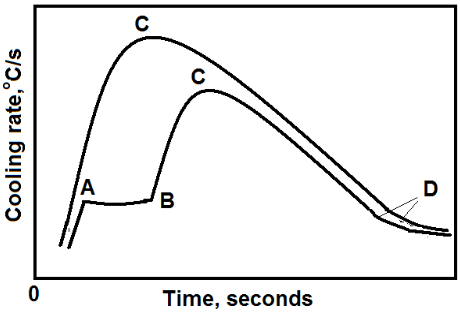

**Fig. 1.** Graph of cooling rate versus time when film boiling is present (ABCD curve) and when film boiling is completely absent (ACD curve): AB is film boiling; C is maximal cooling rate; D is cooling rate at the beginning of convection cooling mode

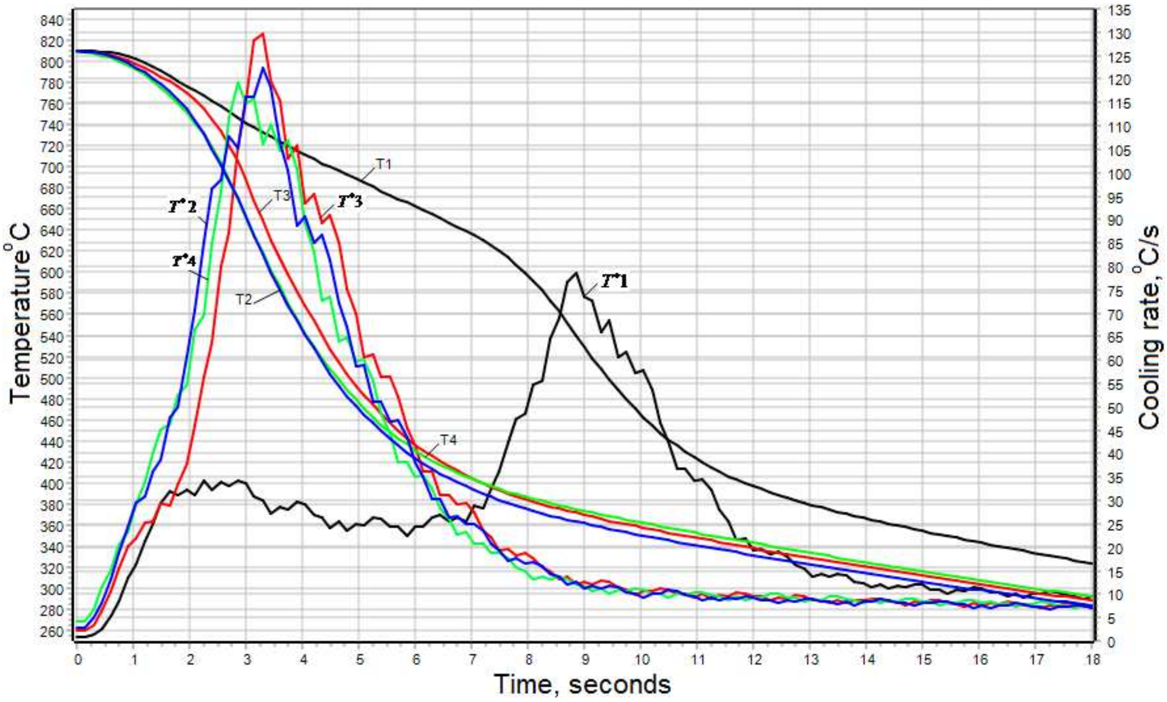

**Fig. 2.** Temperature T and cooling rate $T^*$ at the center of Inconel 600 probe 10 mm in diameter and 30 mm long versus time when quenching in oil I-20A at 50 °C: 1 – no additives at all; 2–7 % of PIB 950; 3–5 % of PIB 1300; 3–4 % of PIB 2400

**Fig. 2** shows absence of film boiling when mineral oil I-20A at 50 °C contains 7 % of PIB 950, 5 % of PIB 1300, or 3 % of PIB 2400 additives. Such concentrations provide the same surface tension and the same dynamic viscosity for every quenchant without the additive. It is counter-intuitive that film boiling disappears with the addition of the PIB additive when surface tension and dynamic viscosity remain unchanged. Based on early investigations [3, 4], the authors believed that one reason for the absence of film boiling is an insulating layer of the additive formed immediately after immersion of the heated high temperature probe into the oil I-20A containing PIB. In the present paper, the authors provide additional support for their thesis by providing appropriate calculations and direct video observation.

## 2. Materials, method and experimental procedure

The main premise of our experiments is as follows: if creation of insulating layer on the surface of probe during quenching in oils containing additives takes place, then increase of PIB in the oil should give a corresponding increase in the heat transfer coefficients (HTCs) at first due to switching from film boiling to nucleate boiling process and then decrease by a smaller amount due to thermal resistance increase caused by thicker insulating layer. Focusing on this idea, the authors used for testing different kinds of oils, I-8A, I-12A, I-20A, and an Inconel 600 cylindrical probe 10 mm in diameter and 30 mm long. The thermal conductivity and diffusivity of Inconel 600 are provided in **Table 1** [5]. A thermocouple was located at the center of the probe. Cooling curves and cooling rates data were recorded by computer. To solve the inverse thermal problem correctly and see the possible insulating layer formation, additional information was collected using video camera for observation. These additional data allowed us to determine more the boundary conditions during quenching in different types of oils [5].

**Table 1**

Thermal conductivity and diffusivity of Inconel 600 material versus temperature

| Temperature, °C | Thermal conductivity, W/mK | Thermal diffusivity, $a\times10^{-6}$ m$^2$/s |
|---|---|---|
| 100 | 14.2 | 3.7 |
| 200 | 16 | 4.1 |
| 300 | 17.8 | 4.5 |
| 400 | 19.7 | 4.8 |
| 500 | 21.7 | 5.1 |
| 600 | 23.7 | 5.4 |
| 700 | 25.9 | 5.6 |
| 800 | – | 5.8 |
| 900 | – | 6.0 |

The experimental data, cooling time, cooling rate and temperature at the core of Inconel 600 probe 10 mm in diameter and 30 long when quenching in solutions of PIB 2400 in oil I-20A at 50 °C, are provided in **Table 2.**

**Table 2**

Cooling time, cooling rate and temperature at the core of Inconel 600 probe 10 mm in diameter and 30 long when quenching in solutions of PIB 2400 in oil I-20A at 50 °C.

| Measurements | Symbols | Concentration, % wt | | | | | |
|---|---|---|---|---|---|---|---|
| | | 0.0 | 0.5 | 1.0 | 1.5 | 2.0 | 3.0 |
| Cooling time from 850 °C to 600 °C in sec. | $\tau_{600}$ | 8.1 | 7.8 | 5.7 | 5.3 | 4.7 | 3.5 |
| Cooling time from 850 °C to 400 °C in sec. | $\tau_{400}$ | 12.0 | 11.3 | 9.2 | 8.8 | 8.1 | 7.2 |
| Cooling time from 850 °C to 200 °C in sec. | $\tau_{200}$ | 37.3 | 36.3 | 34.7 | 35.7 | 34.5 | 34.7 |
| Maximal cooling rate in °C/s | $v^{max}$ | 74.7 | 88.4 | 103.4 | 111.8 | 108.8 | 119.4 |
| Core temperature of the probe at maximal cooling rate in °C | $T_{core}$ | 551 | 550 | 584 | 617 | 635 | 669 |
| Cooling rate when core temperature is 300 °C in °C/s | $v_{T=300}$ | 8.6 | 8.2 | 8.0 | 7.7 | 8.1 | 8.3 |

Similar experimental data, cooling time, cooling rate and temperature at the core of Inconel 600 probe 10 mm in diameter and 30 long when quenching in solutions of PIB 2400 in oil I-8A at 50 °C, are provided for low concentration of PIB in oil I-8A in **Table 3** and for elevated concentration in **Table 4**.

**Table 3**

Cooling time, cooling rate and temperature at the core of Inconel 600 probe 10 mm in diameter and 30 long when quenching in solutions of PIB 2400 in oil I-8A at 50 °C

| **Measurements** | **Symbols** | **Concentration, % wt** | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | **0** | **0.5** | **1.0** | **2.0** | **3.5** | **10.0** | **14.0** |
| Cooling time from 850 °C to 600 °C in sec. | $\tau_{600}$ | 9.4 | 8.7 | 6.7 | 5.5 | 5.4 | 5.4 | 5.8 |
| Cooling time from 850 °C to 400 °C in sec. | $\tau_{400}$ | 12.9 | 11.4 | 8.9 | 7.7 | 7.5 | 7.9 | 8.5 |
| Cooling time from 850 °C to 200 °C in sec. | $\tau_{200}$ | 32.0 | 30.3 | 28.4 | 27.7 | 27.9 | 32.5 | 34.5 |
| Maximal cooling rate in °C/s | $v^{max}$ | 78.5 | 94.2 | 108..2 | 123.2 | 120.7 | 120.2 | 106.4 |
| Core temperature of the probe at maximal cooling rate in °C | $T_{core}$ | 477 | 482 | 564 | 598 | 595 | 581 | 548 |
| Cooling rate when core temperature is 300 °C in °C/s | $v^{T=300}$ | 10.5 | 11.0 | 10.2 | 9.2 | 9.4 | 7.7 | 7.6 |

**Table 4**

Cooling time, cooling rate and temperature at the core of Inconel 600 probe 10 mm in diameter and 30 long when quenching in solutions of PIB 2400 in oil I-8A at 50 °C (high concentration)

| **Measurements** | **Symbols** | **Concentration, % wt** | | | | |
|---|---|---|---|---|---|---|
| | | **0.0** | **14.0** | **16.0** | **19.0** | **21.5** |
| Cooling time from 850 °C to 600 °C in sec. | $\tau_{600}$ | 9.4 | 5.8 | 5.5 | 6.3 | 6.5 |
| Cooling time from 850 °C to 400 °C in sec. | $\tau_{400}$ | 12.9 | 8.5 | 8.4 | 9.5 | 10.1 |
| Cooling time from 850 °C to 200 °C in sec. | $\tau_{200}$ | 32.0 | 34.5 | 36.1 | 37.4 | 38.6 |
| Maximal cooling rate in °C/s | $v^{max}$ | 78.5 | 106.4 | 96.2 | 94.2 | 87.5 |
| Core temperature of the probe at maximal cooling rate in °C | $T_{core}$ | 477 | 548 | 580 | 570 | 595 |
| Cooling rate when core temperature is 300 °C in °C/s | $v^{T=300}$ | 10.5 | 7.6 | 6.7 | 6.0 | 6.2 |

Using the data shown in the above **Tables 1–4,** one can calculate the heat transfer coefficients (HTCs) depending on concentration of PIB in oil. The method of calculation is known and it consists in Kondratjev number Kn evaluation which is [6, 7]:

$$\mathrm{Kn} = \frac{vK}{a\left(T - T_m\right)}. \tag{1}$$

There is an universal correlation between Kondratjev number Kn and generalized Biot number $\mathrm{Bi}_V = \frac{\alpha}{\lambda} K \frac{S}{V}$, which can be found in tables published in literature [6, 7].

Knowing the generalized Biot number $\mathrm{Bi}_V$, one can calculate the heat transfer coefficient from the equation (2) [7]:

$$\alpha = \frac{\lambda \mathrm{Bi}_V}{K\left(S/V\right)}. \tag{2}$$

For the Inconel 600 probe of 10 mm diameter and 30 mm long the following parameters are true:

$$K = 4.127\times10^{-6}\,m^2;\quad K(S/V) = 1.926\times10^{-3}\,m;$$

K is a Kondratjev form factor in $m^2$; S is surface in $m^2$; V is volume in $m^3$; $\lambda$ is thermal conductivity in W/mK; a is thermal diffusivity in $m^2/s$ which are provided in **Table 4.**

Initial heat flux density is calculated by equation (3) which was discussed and used for determining $q_{in}$ by author [2, 3], which should be compared with the critical heat flux densities [8–11]:

$$q_{in} = \frac{k_1 \lambda Kn}{\left(1 + 2\frac{\delta}{l}\frac{\lambda}{\lambda_{coat}}\right) l}\left(\bar{T}_{rg} - T_S\right), \tag{3}$$

$$\frac{V}{KS} = \frac{k_1}{l};\quad l = \frac{L}{2},$$

where $q_{in}$ is the initial heat flux density in $W/m^2$; $k_1$ is the coefficient depending on the form of a steel part; $\lambda$ is the thermal conductivity of steel in W/mK; $\lambda_{coat}$ is the thermal conductivity of the coating (polymeric layer); Kn is the dimensionless Kondratjev number; $\delta$ is the thickness of the polymeric layer in m; l is the radius or half of the thickness of the plate in m; $\bar{T}_{rg}$ is the average temperature at the moment of establishing a regular thermal process; $T_S$ is the saturation temperature; S is the surface in $m^2$; V is the volume in $m^3$; K is the Kondratjev form factor coefficient in $m^2$.

In the literature were published discussions [12–14] showing that critical heat flux densities should be studied carefully to create the possibility of reducing distortion in steel parts during quenching [15–18].

As one can see from Eq. (3), thickness of insulating layer $\delta$ insignificantly affects initial heat flux but does prevent film boiling when $q_{in} < q_{cr1}$. Transition from film boiling to nucleate boiling processes versus concentration of PIB-2400 in mineral oil I-8A at 50 °C is shown in **Fig. 3**.

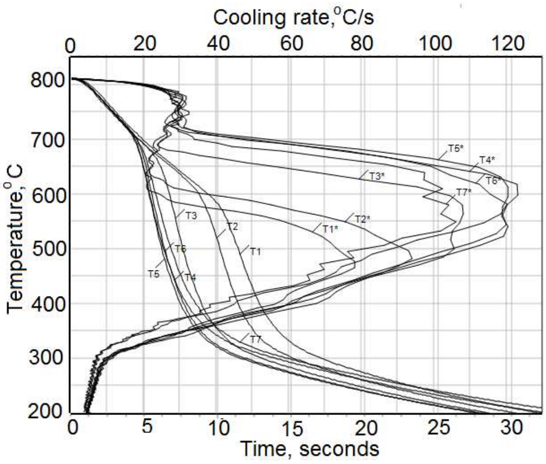

**Fig. 3.** Temperature T and cooling rate $T^*$ versus time at the core of Inconel 600 probe during quenching in solution of PIB-2400 in oil I-8A at 50 °C, % wt: 1 – 0; 2 – 0.5; 3 – 1.0; 4 – 2.0; 5 – 3.5; 6 – 10; 7 – 14

## 3. Discussion of results of experiments

Researchers investigating cooling capacity of quenchants focused on the additives that changed surface tension on the boundary of a solid surface to the liquid [19–21].

The results of experiments presented in **Table 2–4 and Fig. 2** allow calculation of heat transfer coefficients (HTCs) depending on concentration of PIB in mineral oil. If our hypothesis on the existence of an insulating layer on the surface of quenched steel parts is true, then behavior of HTCs should demonstrate this phenomenon. At the beginning HTCs should increase considerably achieving maximum value at optimal concentration of PIB in oil and then very slowly decrease. Such behavior is explained by preventing film boiling process when thickness of insulating layer is optimal which lowers the initial heat flux density below $q_{cr1}$ preventing film boiling and considerably increasing HTCs. Further increase of PIB concentration in mineral oil should decrease HTC very slowly due to slow increase the thickness of insulating layer and as a result slow the increase in thermal resistance. Results of calculations of HTCs at the moment when cooling rate is maximal versus concentration of PIB in oil are provided in **Table 5** which were determined using equations (1) and (2).

**Table 5**
Heat transfer coefficients in W/m²K versus concentration of PIB-2400 in oils I-20A and I-8A at 50 °C

| Type of oil | Symbols | Concentration, % wt | | | | | |
|---|---|---|---|---|---|---|---|
| | | 0.0 | 1.0 | 1.5 | 2.0 | 3 | 14 |
| I-20A at 50 °C | $\alpha_{I-20A}$, W/m²K | 1530 | 2135 | 2030 | 1993 | 2120 | 1770 |
| I-8A at 50 °C | $\alpha_{I-8A}$, W/m²K | 1950 | 2360 | 2410 | 2460 | 2400 | 2210 |

The results of calculations shown in **Table 5** support our thesis that PIB should create a micro layer of insulation to provide a slow increasing and then a slow decreasing of the HTCs versus PIB concentration. To test our hypothesis as true, the video observation was done and showed the hypothesis is correct, as is illustrated in **Fig. 3.**

*a* *b* *c*

**Fig. 4.** Film boiling *a* and creation of insulating surface layer *b* during quenching of cylindrical probe 12.5 mm in diameter in mineral oil I-20A which were captured by video camera: *a* – film boiling at the moment 1.5 sec when core temperature is 780 °C; *b* – film boiling is almost finished at the moment 3.2 sec when core temperature is 720 °C and formation of insulating layer starts; *c* – nucleate boiling process on the surface of insulating layer during cooling in oil I-20A at 50 °C at the moment 5.8 sec when core temperature is 525 °C

In our opinion, the additives of PIB accelerate drastically the formation of a thicker insulating layer that decreases initial heat flux density and prevents film boiling completely which will lower part distortion from heat treat quenching.

**Fig. 4, *a*** shows film boiling during cooling the probe 12.5 mm in diameter in oil I-20A, **Fig. 3, *b*** shows film boiling when it is almost finished; at the moment 3.2 sec, core temperature is 720 °C and formation of insulating layer starts shows. Nucleate boiling process on the insulated surface during cooling in solution 3 % PIB in oil I-8A at 50 °C and **Fig. 3, *c*** illustrates transient nucleate boiling process on the insulated surface layer created just by I-20A oil. Insulation layer is clearly visible.

Now, there is no doubt that the reason for elimination of film boiling during quenching is a micro insulating layer from the PIB additive. It should be emphasized that future investigators should evaluate the thickness and thermal properties of the insulating layers needed for initial heat fluxes determination. Also, authors believe that it will be possible to combine film boiling elimination with part corrosion prevention.

As a rule, film boiling elimination was provided by changing the physical properties of the liquid, especially surface tension values. The proposed approach of PIB additions is a new direction in the elimination local and full film boiling that will decrease distortion of steel parts after quenching in liquid oil media.

## 4. Conclusions

1. There is an optimal concentration of PIB in mineral oil I-20A for the elimination of film boiling caused by creating insulating surface layer of the additive. It decreases initial heat flux density which is less than the first critical heat flux so the local film boiling and full film boiling are absent.

2. The PIB additives to minerals oils I-12A and I-8A decrease significantly the duration of film boiling. Presence of a short period of film boiling in oils I-12A and I-8A is explained by their lower critical heat flux densities as compared with the mineral oil I-20A. The PIB additives were unable to decrease enough initial heat flux densities to prevent completely film boiling, but they reduced duration of film boiling considerably.

3. As expected, increasing additives PIB in oils firstly increase considerably the HTCs and then decrease HTCs by a lesser amount during nucleate boiling process caused by creating a thicker insulating layer that increases thermal resistance.

4. The proposed approach for additions of PIB by the authors [2–4], for decreasing initial heat flux densities by creating a surface insulating layer to limit the process of film boiling, is a very promising method to decrease distortion taking place during quenching of steel parts in liquid media.

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