Transient Nucleate Boiling Process Used for Obtaining Super Strong Carbon Steels and Irons

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Abstract — Based on self-regulated thermal process, in the paper four types of thermomechanical treatments are considered. The first is a high temperature thermomechanical treatment (HTTMT) followed by complete martensitic transformation. The second is a low temperature thermomechanical treatment (LTTMT) plus martensitic transformation. The third is the high and low temperature thermomechanical treatment (HTTMT and LTTMT) plus martensitic transformation. And the last includes HTTMT and LTTMT plus bainitic transformation to obtain super strong and ductile materials. It is shown in the paper that listed technologies are enough intensive to obtain very strong and ductile materials using plain high carbon steels. A detailed consideration of all processes in the paper will motivate engineers to perform mentioned technologies in forging shops to receive super strong and ductile materials without costly alloying that saves energy and alloying elements. The paper discusses the opportunity of preventing martensite transformation to receive fine and nano-bainitic microstructure during intensive quenching. A hypothesis is forwarded that explains possible technology used in 8th and 9th centuries in the Middle East to manufacture Damascus steel. The secret of Damascus steel could be the duration of transient nucleate boiling process needed for preventing martensite transformation during forging of steel.

Keywords — boiling process, thermomechanical treatment, martensitic and bainitic transformation, super strong materials.

I. INTRODUCTION

When film boiling during quenching in liquid media is absent, the self-regulated thermal process is established in 1.5..2 seconds [1]. The discovered phenomenon is a basis for designing super strong materials and cardinally simplifies temperature fields calculation. It allows also accurately calculate transition from nucleate boiling process to convection. As known, some authors during recipes development for intensive quenching processes don't calculate transition from nucleate boiling process to convection. They, as a rule, are using average values of heat transfer coefficients which cannot provide the best results of recipes calculation. Such approach is approximate even for intensive quenching and results in essential error when hardening metal in still liquids where heat transfer coefficients differ from 10 to 100 times. Averaging doesn't work here. Also, the unsolved problem is the transition from experimental data collected for standard probe to data of real steel parts. As the first step, this problem was solved in a recently published paper [2]. As known, heat transfer coefficients (HTCs) during quenching steel parts in water and water salt solutions are approximately calculated from the well-known dimensionless equation of similarity [3]. Such equations cannot be used for water polymer solutions when the surface polymeric layer is formed creating multilayer domain. Based on self-regulated thermal processes and huge number of experiments, Intensive Technologies Ltd (ITL), Kyiv, Ukraine, has developed methodology and software for cooling time calculation during quenching forgings and machine components of any form and size in different liquid media. All of this made the possibility to perform high and low temperature thermomechanical treatment and prevent martensite transformation in conditions when cooling is accelerated. Taking these achievements into account, the current paper discusses the possibility of obtaining super strong and ductile materials made of plain carbon steels and irons.

II. NUCLEATE BOILING AND CONVECTION TAKING PLACE DURING QUENCHING

As known, the self-regulated thermal process allows regulation of the surface temperature of quenched steel parts by adjusting saturation temperature via varying concentrations of water salt solution or pressure in quench tank. Such regulation is performed in conditions when Biot number tends to infinity, *i.e.*, $Bi_v \rightarrow \infty$. It is assumed that film boiling process is completely absent and that can be done by maximizing critical heat flux densities or using the optimal concentration of inverse solubility polymers that create a

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thin insulating layer. The insulating layer decreases initial heat flux density dropping it below its critical value and by this way eliminates film boiling process. Parabolic non- linear heat conductivity equation (1) includes internal thermal sources $Q(T)$ which are generated by phase transformation inside the quenched steel part [3].

$$c\rho\frac{\partial T}{\partial \tau} = \text{div}[\lambda(T)\text{grad}T] + Q(T) \quad (1)$$

Non- linear boundary condition (2) for the first time was formulated by authors in [4].

$$\left[\frac{\partial T}{\partial r} + \frac{\beta \odot^m}{\lambda} (T - T_s)^m \right]_{r=R} = 0 \quad (2)$$

Since during heating to austenitizing temperature its field in steel part is uniform, the initial condition is written as:

$$T(r, 0) = T_o \quad (3)$$

After transient nucleate boiling process is finished, boundary condition has a normal form and is the third type of a boundary condition (4):

$$\left[\frac{\partial T}{\partial r} + \frac{\alpha}{\lambda} (T - T_m) \right]_{r=R} = 0 \quad (4)$$

At the end of transient nucleate boiling process and beginning of convection, the initial condition (5) is written as:

$$T(r, \tau_{nb}) = T(r, \tau) \quad (5)$$

The time of such transition is evaluated from the equalness (6) of heat flux densities q_{nb} and q_{conv} , *i.e.*,

$$q_{nb} = q_{conv} \quad (6)$$

Especially important is a core temperature of steel part at the moment of transition from boiling process to convection. Nowadays, engineers, as a rule, use average HTC considering nucleate boiling and convection as one process. Such consideration can be approximately used for intensive technologies where HTCs during nucleate boiling and convection differ insignificantly between each other. As it was mentioned above, during quenching in still or low agitated liquid media, difference between convection and nucleate boiling HTCs can differ 100 times and more that generates big errors and makes recipes calculation invalid. Intensive Technologies Ltd (ITL) has elaborated software for cooling time calculations and proposed to use resonance effect for preventing film boiling process during quenching in liquid media [5].

To be more specific, below are provided convective HTCs depending on pressure and temperature of a liquid. As known, the heat transfer coefficient during convection is calculated from well-known equation (7) [6]:

$$\alpha_{conv} = 0.135 \cdot \left(\frac{g\beta\Delta T}{av} \right)^{1/3} \quad (7)$$

Its value in still condition of liquid depends on pressure which increases temperature difference ΔT (see (7) and Table I).

TABLE I: CONVECTIVE HEAT TRANSFER COEFFICIENTS DEPENDING ON PRESSURE AND WATER TEMPERATURE

P, MPa	Water 10°C	Water 20°C	Water 30°C
	$\alpha_{conv}, \text{W} / \text{m}^2 \text{K}$	$\alpha_{conv}, \text{W} / \text{m}^2 \text{K}$	$\alpha_{conv}, \text{W} / \text{m}^2 \text{K}$
0.1	548	640	1015
0.2	586	690	1105
0.3	609	719	1156
0.4	625	740	1196
0.5	638	756	1223
1.0	677	806	1310

It should be noted here that self-regulated thermal process establishes in 1.2–1.5 s for any size and form of steel part and surface temperature during boiling process maintains at the level of saturation temperature (see Table II) [7]. According to authors [3], HTC during nucleate boiling vary within 10,000 W/m²K and 200,000 W/m²K. It means that it doesn't make sense to use average values, summarizing nucleate boiling and convection, for cooling time calculation during quenching steel in liquid media.

III. STEEL QUENCHING IN LIQUID MEDIA UNDER PRESSURE

Quenching under pressure allows adjusting the surface temperature during nucleate boiling to delay or accelerate martensite transformation. Also, elevated pressure increases the first critical heat flux density that in many cases eliminates film boiling process. Increasing pressure to 1.0 MPa, one can keep the surface temperature at the level of 200°C (see Table II).

TABLE II: SATURATION TEMPERATURE T_s VERSUS PRESSURE P FOR PURE WATER

P, MPa	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
$T_s, ^\circ\text{C}$	99.6	120.2	133.5	143.6	151.8	158.8	165	170.4	175.4	180

As known, average surface temperature during nucleate boiling process consists saturation temperature and an overheating of a liquid. The initial overheating at the beginning of a self-regulated thermal process is evaluated from (8) while the temperature at the end of nucleate boiling is evaluated from (9) [3]:

$$\vartheta_I = 0.293 \cdot \left[\frac{2\lambda(\vartheta_o - \vartheta_I)}{R} \right]^{0.3} \quad (8)$$

$$\vartheta_{II} = 0.293 \cdot [\alpha_{conv}(\vartheta_{II} + \vartheta_{uh})]^{0.3} \quad (9)$$

Taking these facts into account, it is possible to prevent martensite transformation during intensive quenching by adjusting pressure in a system shown in Fig. 1.

Fig. 1. Basic scheme of the automated process of steel quenching in water and aqueous solutions under pressure [3]: 1, tray; 2, aperture for pumping in compressed air; 3, mobile piston (cover); 4, case of the quench tank; 5, the part to be quenched; 6, solenoid for fixing the initial time of transformation of austenite into martensite; A, the amplifier of a signal of the martensite start; R, relay of current; G, driving mechanism; I, starting position; II, work position.

As an example, for quenching under pressure was used cylindrical probe 50 mm diameter and 150 mm long with Kondratiev coefficient $K = 103.17 \times 10^{-6} \text{ m}^2$. Average thermal conductivity of steel is 23 W/mK and its thermal diffusivity is $5.4 \times 10^{-6} \text{ m}^2/\text{s}$. As seen from Table III, surface temperature of probe during its quenching under normal pressure maintains at the level of 114 °C while during quenching under pressure 0.7 MPa surface temperature of probe maintains at the level of 190 °C (see Fig. 2 a, b).

TABLE III: CHARACTERISTICS OF COOLING PROCESS RELATED TO CYLINDRICAL PROBE 50 MM DIAMETER AND 150 MM LONG WHEN QUENCHING FROM 850 °C IN WATER AT 20 °C UNDER PRESSURE

P, MPa	Initial surface boil temperature $T_I, ^\circ\text{C}$	Surface temperature at the end of boiling $T_{II}, ^\circ\text{C}$	Nucleate boiling duration τ_{nb}, s	Core temperature at the end of boil $T_{core}, ^\circ\text{C}$	Maximal core cooling rate $\dot{\gamma}_{\max}, ^\circ\text{C/s}$	Core cooling rate at the end of boiling, s
0.1	120	107	68	200	18.5	3.5
0.3	153.6	140.5	58	270	17.8	4.7
0.5	172	159	54	300	17.1	5.2
1.0	200.5	188	49	350	17	6

a)

b)

Fig. 2. Surface and core cooling curves versus time when quenching cylindrical probe 50 mm diameter and 150 mm length in water at 20°C under normal (0.1MPa) and elevated (0.7 MPa) pressure.

Note, that maximal cooling rate changes insignificantly with changing pressure while cooling rate at the end of nucleate boiling does.

Fig. 3. Cooling rate curve versus time when quenching cylindrical probe 50 mm diameter and 150 mm length in water at 20°C under normal (0.1MPa) pressure.

IV. HIGH AND LOW TEMPERATURE THERMOMECHANICAL TREATMENT

High and low temperature thermomechanical treatment was used in the practice a long ago [8]-[10]. It improves significantly strength of steel and its plastic properties (see Table IV and Fig. 4, a). Table IV provides only results of high temperature thermomechanical treatment (HTTMT).

TABLE IV: IMPROVEMENT OF MECHANICAL PROPERTIES OF AISI 1040 STEEL BY HIGH TEMPERATURE THERMOMECHANICAL TREATMENT AFTER TEMPERING AT 200 °C [9], [11]

Technology	R_m , MPa	$R_{p0.2}$, MPa	A (%)	Z (%)	a_k , J/cm ²
Conventional	1422	1246	2	16	30
DFIQ	1972	1570	7	40	35

Fig. 4. Schemes of performing of high and low temperature thermomechanical treatment resulting in martensitic microstructure (a) and that resulting in bainitic microstructure (b).

As seen from Table IV, ultimate strength R_m of AISI 1040 after high temperature thermomechanical treatment increased for 38%, yield strength $R_{p0.2}$ increased for 26%, elongation increases for 350% and impact strength a_k increases for 17%. As a rule, during conventional quenching, strength increase decreases plastic properties of a material. High temperature thermomechanical treatment increases both strength and plastic properties of steel. Similar improvements of mechanical and plastic properties were observed by authors [12] who performed high temperature thermomechanical treatment in condition of intensive cooling and called it DFIQ process. It means direct intensive quenching after forging. As seen from Table IV, even one cycle of HTTMT shows essential progress in mechanical properties improvement. More benefits one could expect if a chain of treatments is performed.

- Delaying martensite transformation by self-regulated thermal process in condition $Bi_V \rightarrow \infty$.
- High temperature thermomechanical treatment (HTTMT) plus martensitic transformation.
- Low temperature thermomechanical treatment (LTTMT) plus martensitic transformation.
- High and low temperature thermomechanical treatment (HTTMT and LTTMT) plus martensitic transformation.
- HTTMT and LTTMT plus bainitic transformation to obtain super strong and ductile materials.

The last is the most beneficial since, according to author [13], bainitic transformation provides better mechanical and plastic properties of material. A scheme of performing an intensive HTTMT and LTTMT plus bainitic transformation to receive super strength and ductility steel is shown in Fig. 4 b). The quench system (see Fig. 3) can be used for performing HTTMT and LTTMT plus bainitic transformation for different steels containing carbon from 0.8 % C to 1.8 %C (see Table V).

TABLE V: MARTENSITE START TEMPERATURE M_s VERSUS OF CONTENT OF CARBON IN STEEL IN %, WT

C, % wt	0.8	1.0	1.1	1.2	1.3	1.4	1.5	1.6
M_s , °C	250	214	200	180	160	145	120	100

The mentioned steels have low enough martensite start temperature M_s to prevent martensite transformation during intense quenching of forgings using appropriate pressure in the system (Fig. 3).

As a basis for performing HTTMT and LTTMT plus bainitic transformation is universal equation (10) which is true for different forms and sizes of steel parts [14]:

$$\tau_{nb} = \bar{\Omega} k_F \frac{D^2}{a} \quad (10)$$

Here τ_{nb} is duration of transient nucleate boiling process in s; $\bar{\Omega}$ is dimensionless value which depends on initial heating temperature and convective Biot number Bi ; k_F is form coefficient; D is thickness in m; a is thermal diffusivity of steel in m^2/s .

Fig. 5. Dimensionless value Ω versus Biot number Bi [14].

The value Ω is true for initial temperature 850 °C. When quenching forgings from 950 °C, the mentioned value Ω increases for 1.7%.

V. DISCUSSION

It is said that ancient swords and knives (9th century) were made of Woodz (Indian), Pulad (Persian), Fuladh (Arabic), Bulat (Russian) and Binite (Chinese). Damascus steel was the forged steel of the blades of swords smithed in the Near East from ingots of Woodz steel [15]. It is also said that ancient technology was lost and nowadays engineers are trying to duplicate the “Damascus Steel”. As known, the “Damascus steel” is the plain ultra-high carbon steel subjected to special forging and quench process resulting in mentioned above super - sharpness and strength of cutting tools and knives. The author of current paper pays the main attention to possible ancient recipes of achieving super strong materials and proposes new technologies for performing high-temperature and low temperature thermomechanical treatment to be widely used in forging shops [16]. This is important for the practice since nowadays many industries are using ductile iron for manufacturing different kinds of tools and machine components. Ductile iron in many aspects is similar to ultra- high carbon steel. The paper discusses forging and quench process in liquid media under pressure which varies within 01-1 MPa. Special attention is paid to delaying martensite transformation to achieve after forging and quenching fine bainitic or nano-bainitic microstructure. Investigations of author [13] showed that bainitic microstructure has better mechanical and plastic properties as compared with martensite. Physics of quench process for possible ways of manufacturing the Damascus, probably, can be considered as the high and low temperature thermomechanical treatment with delaying martensite transformation. It is thought that the Woodz was heated to temperature that exceeds A_{c1} and then forged by hammer to certain shape and quenched in water or water salt solution until nucleate boiling is finished. Since for the high carbon steel martensite start temperature is below saturation temperature, martensite transformation was completely delayed that allowed the high and low temperature thermomechanical treatment. Such operation was repeated several times that included the high and low temperature thermomechanical treatment. Finally, the process was continued via the bainitic transformation to obtain fine or nano microstructure. The secret of Damascus steel could be cooling time interruption (see (10)) and content of carbon in steel that allowed to delay martensitic transformation. Duration of transient nucleate boiling process could be evaluated by hearing the noise of boiling process and feeling the tiny vibration of sword during quenching. The experienced masters could easily produce such process to obtain super strong swords with the high elastic properties and wear resistance. At present time, special attention should be paid to elimination film boiling processes by increasing critical heat flux densities [17], [18], and

[19] or decreasing initial heat flux densities. Sonar control system can help to eliminate film boiling processes during quenching [20]. The results of investigations presented in this paper can be used by forging shops to produce super strong materials from high carbon steels and irons.

VI. CONCLUSIONS

1. In the paper the self-regulated thermal process (SRTP) was used for designing super strong and ductile materials. The SRTP is an intensive cooling, and its peculiarity consists in possibility of adjusting surface temperature in condition when $Bi_v \rightarrow \infty$. This fact allowed performing different types of thermomechanical treatment with complete preventing martensite transformation during very intensive cooling.
2. A tendency of increasing strength of steel and its plastic proper during high temperature thermomechanical treatment is shown in the paper.
3. The author of the paper proposes to perform high and low temperature thermomechanical intensive treatment combined with the fine and nano – bainitic transformation to attain super strong and plastic materials.
4. The hypothesis is forwarded by author on possibility of use of thermomechanical treatments for duplicating the Damascus steel.
5. To make any quench process successful, a package of developments (appropriate quenchant, software for emitter agitation and cooling time interruption, a system for quality quench control) should be used together when applying new technologies into practice.

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