DOI: https://doi.org/10.24425/amm.2023.145466

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STUDY OF THE EFFECT OF INTENSITY OF VIBRATIONS AND TEMPERATURE ON THE COOLING CHARACTERISTICS OF QUENCHING MEDIA

Quenching technology requires the use of media with different cooling intensities and various shapes of cooling curves that show different particularities compared to that of conventional media such as water, oil, or emulsions. The use of synthetic quenching media is relatively new and also has multiple advantages such as non-flammability, safety in use and low cost. In this study, the cooling media tested was obtained by mixing 2 wt% carboxymethyl cellulose with 2 wt% NaOH in one litter of water. Moreover, three different temperatures (20°C, 40°C and 60°C) of the quenching media were evaluated. By dissolution in water, a synthetic solution with low viscosity, surfactant and lubricant was obtained. Because carboxymethyl cellulose is a biodegradable organic material, that is obtained as a by-product in the manufacture of paper, a basic substance with a preservative effect was added. According to this study, both the variation diagram of the heat transfer coefficient and the diagram of the cooling rates, during the cooling stages give important indications regarding the use of a liquid cooling medium for quenching.

Keywords: quenching media; synthetic cooling media; vibrations; heat treatments; carboxymethyl cellulose

1. Introduction

A liquid cooling medium, which changes its state of aggregation during cooling, in order to ensure the structural transformation at hardening, ensures a heat exchange conditioned by the intensity of heat transmission between the hot part and the cooling medium. The heat transfer is governed by the heat transfer coefficient from the metal surface to the cooling medium which is achieved by radiation and conduction [1-4].

Evaporation of the quenching medium influences the heat transfer at high temperatures, mostly, in the first period of cooling.

In the case of liquid cooling media that have a vaporization temperature lower than the surface temperature of the heattreated sample, four stages occur, depending on the temperature difference between the metal surface and the cooling medium [5-7]:

- a) instantaneous cooling of the metal surface, by sudden heating of the liquid medium by approaching the part until the vaporization temperature is reached;
- b) heating, which consists of the formation of a vapor coating on the sample surface and which reduces the heat exchange rate;

- boiling, which occurs by piercing the steam coating and removing it by forming bubbles that will condense after removal from the hot metal;
- d) convection, occurs after the metal surface has a lower temperature than the boiling temperature of the liquid medium.

In the first stage, the heat exchange is intense and short in time, in the second stage, due to the formation of a gas layer, the sample is insulated and consequently, the heat exchange is mainly by radiation, which causes the cooling rate to decrease significantly.

When the steam layer breaks by boiling, the heat exchange intensifies, and the cooling rate increases. After the temperature of the metal surface drops below the boiling point of the liquid, the heat exchange takes place by convection and the cooling rate decreases.

Consequently, the heat transfer coefficient between the metal and the liquid medium varies in high and uneven ranges (the maximum being the boiling temperature), while an important role is played by the degree of agitation of the medium, despite the cooling periods [5,8]. When assessing the cooling process for hardening in the liquid medium, due to numerous influencing factors, it is not enough to take into account only the heat

¹ "GHEORGHE ASACHI" TECHNICAL UNIVERSITY OF IASI, FACULTY OF MATERIALS SCIENCE AND ENGINEERING, PROF. D. MANGERON STREET, NO. 41, 700050, IASI, ROMANIA ² ROMANIAN INVENTORS FORUM, SF. P. MOVILA 3, IASI, ROMANIA

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© 2023. The Author(s). This is an open-access article distributed under the terms of the Creative Commons Attribution-NonCommercial License (CC BY-NC 4.0, https://creativecommons.org/licenses/by-nc/4.0/deed.en which permits the use, redistribution of the material in any medium or format, transforming and building upon the material, provided that the article is properly cited, the use is noncommercial, and no modifications or adaptations are made. transfer coefficient, therefore, a much more complex mechanism will influence the thermal behavior of the metal sample [9]. Accordingly, a relatively simple, experimental parameter, had to be adopted, namely cooling rate.

The variation of the cooling coefficient (α) influences a corresponding variation of the different cooling rates during the cooling stages, presenting a maximum in the area of the boiling temperature of the medium.

Both the variation diagram of the heat transmission coefficient and the cooling rates diagram, during the cooling stages give important indications regarding the use of a liquid cooling medium for quenching [10,11]. The hardening capacity of a liquid medium in the technological process of heat treatment of quenching, for a certain type of steel, can be confirmed only through the hardening test (the Jominy End Quench test).

Knowing the variation of the heat transmission coefficient, and Biot and Fourier criteria, the cooling rate and temperature variation can be calculated at different distances from the cooling contact surface.

Due to the many difficulties in using mineral oils as a medium for quenching steel parts, it was necessary to find more economical and safer solutions, therefore, synthetic cooling media were used for quenching [12-14]. Moreover, another important parameter that can be manipulated to assure the necessary condition for the hardening of steel parts is the agitation of the cooling medium, which will assure an increase in the cooling rate. The same effect can be obtained by vibrations.

The presence of vibration will influence all stages of the cooling process:

- during the heating period, the vibrations will break the vapor film faster, which is beneficial because it shortens the period when the hot part is practically isolated from the media and the cooling takes place under the effect of radiation through the vapor layer surrounding the part;
- during the interval when the cooling medium boils, the vibrations will break the large bubbles that are dangerous for both the operator and the parts, because the cooling is chaotic and can lead to microcracks formation. Therefore,

the presence of vibrations will decrease the volume of bubbles and will increase the homogeneity of the media;

below the boiling temperature, the vibrations will intensify the convection.

Moreover, the intensity of the vibration could influence the quenching parameters, accordingly, low-frequency vibration has a stronger effect on mixing and smoothing the bath compared to high frequencies.

Synthetic cooling media for quenching are very diverse and sometimes contain by-products or wastes resulting from different industries [15]. Carboxymethyl cellulose is a cellulose gum derivative with carboxymethyl groups (-CH2-COOH) that results as a by-product of papermaking [16,17]. In an aqueous solution between 1.5-3.5 wt.% with additives, it can be used as a cooling medium. The additions of carboxyl cellulose in the quenching media aim to improve the cooling properties differentiated on the temperature ranges as well as to preserve the properties of the media because the biological solutions degrade over time.

The aim of this study is to analyze the effect of vibrations and NaOH additives on the modification of the cooling curves of synthetic quenching media such as carboxymethyl cellulose solutions dissolved in water.

2. Experimental results

2.1. Quenching media

In this study, a solution of 2 wt.% carboxymethyl cellulose + 2 wt.% sodium hydroxide dissolute in water was used as quenching media. In Fig. 1, the morphology of the carboxymethyl cellulose can be observed. As can be seen, this addition consists of fibers with different thicknesses.

The experiments were conducted in three different conditions:

 at temperature (T) of 20°C, without vibrations medium or with vibrations at three different frequencies (55 Hz, 110 Hz and 380 Hz);



Fig. 1. Carboxymethyl cellulose microstructure: a) high thickness fibers; b) fine and high thickness fibers

- at a temperature of 40°C, without vibrations medium or with vibrations at three different frequencies (55 Hz, 110 Hz and 380 Hz);
- at a temperature of 60°C, without vibrations medium or with vibrations at three different frequencies (55 Hz, 110 Hz and 380 Hz).

Based on the obtained results, the cooling rates were calculated on cooling intervals.

2.2. Methods

The installation used for the experimental determination of the cooling curves consists of: an air bubbling system; a tube heating system with a control sample made of silver (cylindrical oven with electric resistance); measuring system represented by a y-t recorder (the device used for changing the speed of the recorder), Fig. 2.

The silver specimen with embedded chromel-alumel thermocouple has the following characteristics $\pounds = 13$ [mm]; h = 28 [mm]; S = 1408 [mm²]; m = 39,9 [g]; $\rho_{Ag} = 10,5$ [g/cm³]; $\lambda_{Ag} = 418,5$ [W/m·K].

For the calculation of the thermal transfer coefficient on intervals, the Eq. (1) is used [18]:

$$\alpha_i = \frac{3600 \cdot m \cdot c}{\Delta t_i \cdot S} \ln \frac{T_i - T_0}{T_f - T_0}, \left[W/m^2 \cdot K \right]$$
(1)

where:

- m sample weight, m = 0.0399 [kg];
- c specific heat of silver, c = 0.056 [kcal/kg·grd];
- S sample surface, $S = 0.001408 \text{ [m^2]}$;
- Δt measured time intervals, [s];
- T_i, T_f the initial and final temperature of each interval, [°C];
 - T_o quenching media temperature, [°C].

To calculate the cooling intensity of the studied quenching media, Eq. (2) was used [18]:

$$H^{-1} = \frac{\alpha_g}{2\lambda_{\rm Ag}}, \left[{\rm m}^{-1} \right] \tag{2}$$

where:

 α_g – overall heat transfer coefficient:

$$\alpha_{\sigma} = (\alpha_1 t_1 + \alpha_2 t_2 + \dots + \alpha_n t_n) / \Sigma t_i, [W/m^2 \cdot K]$$

- t_i the time interval between T_i and T_i + 1, [s];
- α_i interval heat transfer coefficient;
- t total cooling time, [s];
- λ_{Ag} coefficient of thermal conductivity of silver.

The silver specimen was heated to austenitizing temperatures of a steel (800°C), after which it was immersed in cooling media for quenching in a calm state, the cooling being done without agitation or vibration with three frequencies.

The synthetic media used for testing was one liter. The tests were repeated to validate the experiment.

3. Results and discussion

An important parameter in changing the intensity and characteristics of cooling is the agitation of the cooling media. There are multiple methods that can assure a turbulent cooling, however, a special category of internal movement of the media, less used, is the vibration of the cooling media with frequencies in all ranges (infrasound, acoustic and ultrasound). Synthetic quenching media are non-flammable, unlike oil, and, usually, they have a lower cooling rate than water, reducing the risk of deformation and cracking.

The first disadvantage of the carboxymethyl cellulose solution is that, when it is not in use, it precipitates on the bottom of the quenching bath. Therefore, it will become a compact layer of sedimentary conglomerates. To reactivate the carboxymethyl cellulose solution in the cooling bath, the media must be agitated by stirring or by vibrations.

TABLES 1, 2 and 3 show the experimental values measured with the experimental installation, and Figs. 3, 6 and 9 show



Fig. 2. The system used to determine the experimental quenching cooling curves: 1 - cylindrical furnace with electric resistance; 2 - silver specimen with embedded chromel-alumel thermocouple; 3 - measuring system with y-t recorder, time base for y-t recorder; 4 - tanks with quenching media

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the curves corresponding to the data in the tables. Moreover, in Figs. 4, 7 and 10 the cooling curves have been plotted.

Figs. 5, 8 and 11 show the variations of the heat transfer coefficient calculated on cooling intervals for the quenching media at different temperatures (20°C, 40°C and 60°C) with and without vibrations. As can be seen from the figures, the heat transfer coefficient has values that vary mainly with the initial



Fig. 3. Cooling curves of water with 2% CMC + 2% NaOH addition at $T = 20^{\circ}$ C, V = 2.5 s/cm



Fig. 4. Cooling rate of water with 2% CMC + 2% NaOH addition at $T = 20^{\circ}$ C, V = 2.5 s/cm



Fig. 5. Variation of heat transfer-coefficient with temperature during cooling in water with 2% CMC + 2% NaOH addition at T = 20°C, V = 2.5 s/cm

temperature, while the presence of vibrations regardless of the frequency has a much lower influence.

Considering the curves at the same temperature, the vibration frequency has a stronger effect when the ambient temperature is 20°C compared to that at 60°C, where the cooling curves at different frequencies are very close.

When the initial temperature of the synthetic cooling media is 20°C, TABLE 1, the cooling curves are more diverse, while the strongest effect is produced by the low-frequency vibrations, i.e., 55 Hz. In general, the presence of vibration in the quenching media has mainly shown an intensification of cooling in the field of minimum stability of the subacute austenite, which favors the obtaining of hard structures.

Another effect observed at ambient temperature $(20^{\circ}C)$ is the uniformity of the cooling, (Figs. 3-5). Due to the vibration, the bubbles film does not form, therefore, the cooling is done without thermal shocks, which will significantly decrease the risk of cracks formation or internal stresses appearance that could conduct to the destruction or deformation of the heat-treated part.

As can be seen from Fig. 3, the cooling media without vibrations at 20°C has a strong variation of non-uniformity in cooling with a false cooling range between 400°C and 300°C temperature, followed by a sudden cooling between 300°C and 200°C.

A similar behavior, but less accentuated, was observed in the curve in Fig. 6, i.e., in non-vibrated environments with an initial temperature of 40°C, TABLE 2.

The cooling stage no longer appears in the non-vibrated environment with an initial temperature of 60°C, TABLE 3.

In vibrated quenching media both those with an initial temperature of 40°C and those with an initial temperature of 60°C (Figs. 9-11) the non-uniformity of cooling is much diminished, which is equivalent in effect to cooling without non-uniformities.

As can be seen from Figs. 4 and 7, the colling media without vibrations exhibit a non-uniform cooling rate, while those with vibrations show a relatively constant cooling rate with only a single maximum.

Fig. 12 shows the cooling intensities calculated for the synthetic media studied with and without vibration. The graph shows that the cooling intensity of the analyzed synthetic medium depends mainly on the temperature being lower at an ambient temperature of 60°C.

The presence of vibrations does not significantly influence the variation of the cooling rate, but it shows a notable change in cooling behavior in different temperature intervals. Accordingly, the vibrations will increase the cooling rate at the temperature ranges between 800°C and 300°C. Also, a constant homogenous

TABLE 1

Cooling characteristics of aqueous solution of 2% CMC + 2% NaOH addition at $T = 20^{\circ}$ C

<i>T</i> [°C]	800	700	600	500	400	300	200	100	70	60
t [s]-0 Hz	0	11.00	11.50	13.50	15.50	21.00	22.13	28.25	58.25	
t [s]-55 Hz	0	9.50	10.25	11.50	13.75	17.88	21.00	25.75		61.00
t [s]-110 Hz	0	15.25	18.38	19.13	21.13	25.00	28.50	35.00	62.50	
t [s]-380 Hz	0	14.00	14.50	15.25	17.75	21.50	24.50	29.75		57.50

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TABLE 2

Cooling characteristics of aqueous solution of 2% CMC + 2% NaOH addition at T = 40°C

<i>T</i> [°C]	800	700	600	500	400	300	200	100	70	60	50
t [s]-0 Hz	0	14.25	29.00	31.75	35.75	41.25	45.00	53.00	62.50		
t [s]-55 Hz	0	10.25	15.00	18.75	24.13	28.00	30.50	38.50		60.00	
t [s]-110 Hz	0	13.38	17.25	20.88	25.75	25.00	31.13	35.00		61.25	
t [s]-380 Hz	0	9.00	13.50	16.88	21.00	25.75	28.00	32.50			62.50

TABLE 3

Cooling characteristics of aqueous solution of 2% CMC + 2% NaOH addition at $T = 60^{\circ}$ C

<i>T</i> [°C]	800	700	600	500	400	300	200	100	90	80
t [s]-0 Hz	0	10.75	21.38	25.00	30.00	35.88	42.50	51.25		61.25
t [s]-55 Hz	0	21.25	26.50	30.50	36.00	40.75	44.75	55.00	62.50	
t [s]-110 Hz	0	23.00	26.50	30.25	35.50	40.13	44.75	55.00		67.50
t [s]-380 Hz	0	15.00	22.75	26.50	31.50	35.75	39.50	48.75		62.50



Fig. 6. Cooling curves of water with 2% CMC + 2% NaOH addition at T = 40 °C, V = 2.5 s/cm



Fig. 7. Cooling rate of water with 2% CMC + 2% NaOH addition at $T = 40^{\circ}$ C, V = 2.5 s/cm



Fig. 8. Variation of heat transfer-coefficient with temperature during cooling in water with 2% CMC + 2% NaOH addition at T = 40°C, V = 2.5 s/cm



Fig. 9. Cooling curves of water with 2% CMC + 2% NaOH addition at $T = 60^{\circ}$ C, V = 2.5 s/cm



Fig. 10. Cooling rate of water with 2% CMC + 2% NaOH addition at $T = 60^{\circ}$ C, V = 2.5 s/cm



Fig. 11. Variation of heat transfer-coefficient with temperature during cooling in water with 2% CMC + 2% NaOH addition at $T = 60^{\circ}$ C, V = 2.5 s/cm

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Fig. 12. The cooling intensity in water with 2% CMC + 2% NaOH addition depending on temperature and vibrations of quenching medium

cooling media will be maintained, i.e., the sedimentation of the carboxymethyl cellulose is diminished.

In other studies [19-20], synthetic quenching media such as aqueous solutions of 2-4% carboxymethyl cellulose, as well as aqueous solutions of polyalkylene glycol were studied as a viable alternative for conventional quenching media such as oil or water which have disadvantages such as very high cooling speeds (water), flammability and high cost price (oil - 23.33).

Carboxymethyl cellulose solutions were also analyzed [19] in a mixture with additives to stabilize and increase the surface tension as well as from the point of view of keeping the cooling properties after multiple uses [20]. It was also observed that moderately used solutions (3-10 uses) have optimal cooling curves and among the most effective modifiers are Na₂SiO₄ and NaOH.

The analyzes were carried out both in a quiet cooling environment and in a bubbling environment to have a more intense heat exchange. It was observed that bubbling has the effect of increasing the non-uniformity of the cooling even on sensitive time intervals for the parts (300-100°C martensitic transformation interval) which led to the idea of using vibrations to emphasize the thermal transfer, but in conditions of uniform thermal shocks dangerous for the integrity of the piece during tempering.

4. Conclusions

At temperatures of 40°C and 60°C, the vibration of the quenching media has a lower effect compared to the effect obtained at 20°C.

The vibration of the quenching media increases the homogeneity of the cooling media by dispersing the carboxymethyl cellulose addition.

Despite the vibration frequency, the vibrated media show an intensification of the cooling rate in the temperature range of minimum stability of the subacute austenite. Therefore, it favors the obtaining of a hardening structure with a smaller amount of residual austenite.

REFERENCES

- J. Barberi, C. Faulkner, D. Scott MacKenzie, Quenching Control and Distortion 9-13, 539-546 (2012).
- [2] J.D. Chen, S.P. Lu, Advanced Materials Research 602-604, 411-414 (2012).
- [3] B. Jez, M. Nabialek, P. Pietrusiewicz, K. Gruszka, K. Bloch, J. Gondro, J. Rzacki, M.M.A.B. Abdullah, A.V. Sandu, M. Szota, K. Jez, A. Salagacki, IOP Conference Series-Materials Science and Engineering 209, 012023 (2017).
- [4] T.D. Blake, Y.D. Shikhmurzaev, J. Colloid Interface Sci. 253, 196-202 (2002).
- [5] A. Buczek, T. Telejko, J. Mater. Process. Technol. 155-156, 1324-1329 (2004).
- [6] S.J. Gokhale, J.L. Plawsky, P.C. Wayner Jr, J. Colloid Interface Sci. 259, 354-366 (2003).
- [7] I. Gradinariu, I. Stirbu, C.A. Gheorghe, N. Cimpoesu, M. Agop, R. Cimpoesu, C. Popa, Materials Science-Poland **32** (4), 578-582 (2014).
- [8] M.J. Barrena-Rodríguez, F.A. Acosta-González, M.M. Téllez-Rosas, Metals 11, 1-17 (2021).
- [9] H.K. Kim, S.I. Oh, J. Mater. Process. Technol. 112, 157-165 (2001).
- [10] J. Hammad, Y. Mitsutake, M. Monde, Int. J. Therm. Sci. 43, 743-752 (2004).
- [11] S.G. Kandlikar, M.E. Sterinke, Int. J. Heat Mass Transfer 45, 3771-780 (2002).
- [12] W.B. Wan Nik, F.N. Ani, H.H. Masjuki, Energy Convers. Manage. 46, 2198-2215 (2005).
- [13] A.A. Zainulabdeen, N.Y. Mahmood, J.H. Mohmmed, IOP Conference Series: Materials Science and Engineering 454, 012053 (2018).
- [14] L. Xinmin, G. Totten, Mater. Perform. Charact. 8, 213-240 (2018).
- [15] J. Hájek, Z. Dlouha, V. Průcha, Metals 11(2), 250 (2021).
- [16] C. Nejneru, A. Grecu, P. Vizureanu, M. Axinte, M.C. Perju, Metalurgia International 18 (6), 33-36 (2013).
- [17] C. Nejneru, P. Vizureanu, A.V. Sandu, A. Grecu, N. Cimpoesu, Revista de Chimie 65 (2), 194-198 (2014).
- [18] P. Vizureanu, Echipamente şi instalaţii de încălzire (eng. Equipment and Installations for heating), 2009, PIM Ed., Iaşi, Romania, ISBN 978-606-520-349-5.
- [19] A. Grecu, C. Nejneru, I. Hopulele, R. Hanu Cimpoeşu, M. Axinte, Research about agitation degree influence of cooling environment on hardening to thermal transfer coefficient between environment sample, Journal of Optoelectronics and Advanced Materials – Symposia, 1 (6), 1071-1076 (2009).
- [20] C. Nejneru, P. Vizureanu, A.V. Sandu, A. Grecu, N. Cimpoesu, Thermal Fatigue of Some Synthetic Hardening Environments with CMC, Revista de Chimie 65 (2), 194-198 (2014).